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INSTITUTE OF METALLURGY AND MATERIALS SCIENCE POLISH ACADAMY OF SCIENCES

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Introduction to Electron Backscatter Diffraction

Electron Backscatter Diffraction (EBSD) is a technique which allows crystallographic information to be obtained from the samples in the Scanning Electron Microscope (SEM)

Project WND-POWR.03.02.00-00-I043/16

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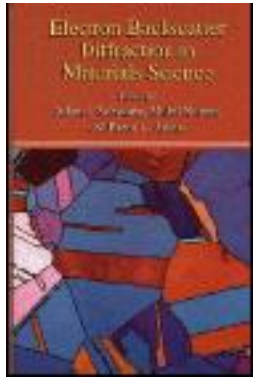
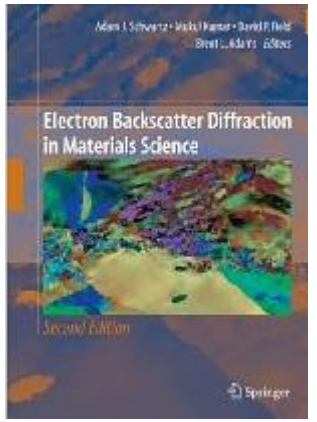
Electron Backscatter Diffraction in Materials Science

Edited by Adam J. Schwartz - *Lawrence Livermore National Laboratory, CA, USA*
Mukul Kumar - *Lawrence Livermore National Laboratory, CA, USA*
Brent L. Adams - *Brigham Young University, Provo, UT, USA*
Kluwer Academic/Plenum Publishers, 2000



Electron Backscatter Diffraction in Materials Science

Editors:
Adam J. Schwartz, Mukul Kumar, Brent L. Adams, David P. Field
Springer, 2009



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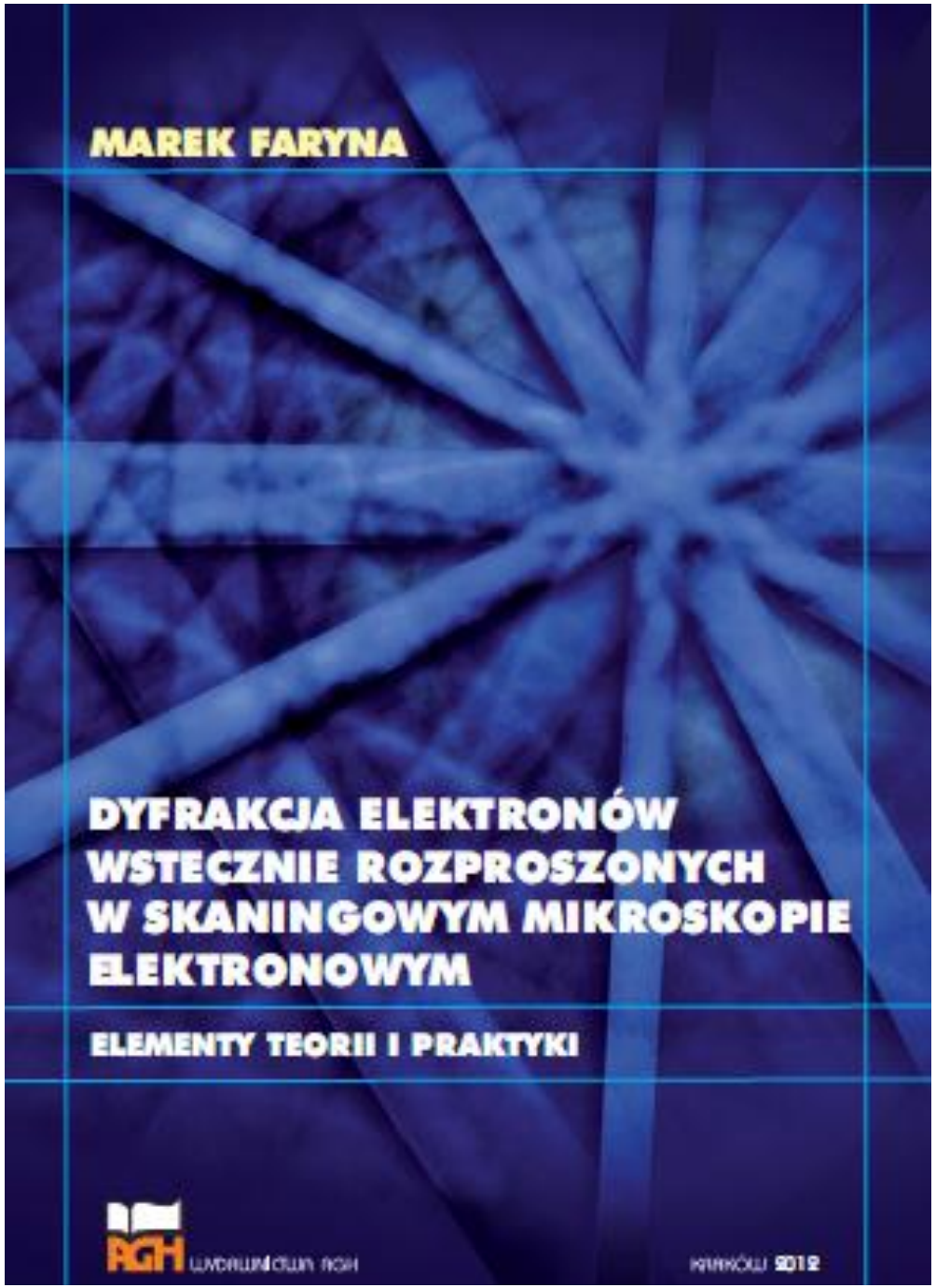

Outline

1. Introduction – a little bit of history
2. EBSD – the basics
3. EBSD – solving the pattern
4. Information available from EBSD
5. A few examples
6. Conclusions

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1. Introduction – a little bit of history

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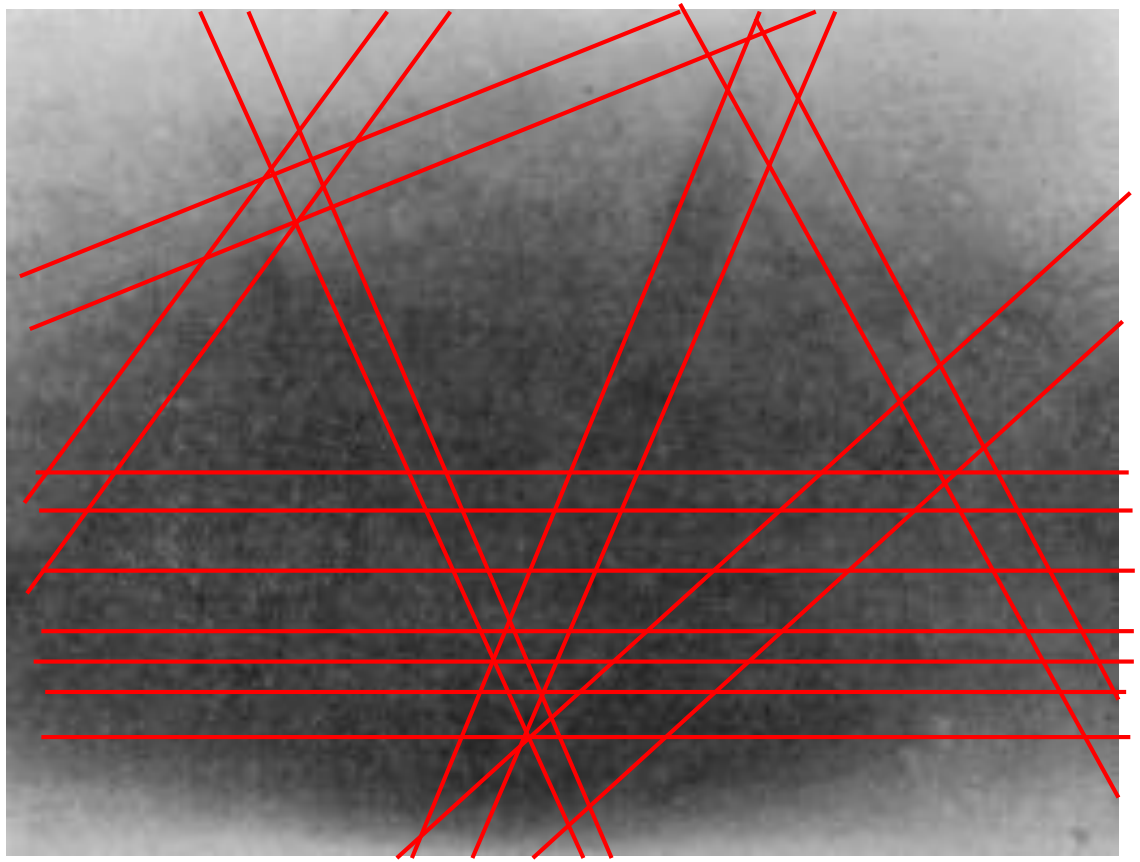
A gas discharge beam of 50 keV electrons was directed onto a cleavage face of calcite at a grazing incidence of 6°. Patterns were also obtained from cleavage faces of mica, topaz, zinc blende and a natural face of quartz.



Shoji Nishikawa and Seishi Kikuchi

The Diffraction of Cathode Rays by Calcite

Proc. Imperial Academy (of Japan) 4 (1928!!!) 475-477

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Point analysis

EBSD - Electron Backscatter Diffraction

EBSP - Electron Backscatter Pattern (J.A.Venables)

BKP - Backscatter Kikuchi Pattern

Scan analysis

COM - Crystal Orientation Mapping

ACOM - Automatic Crystal Orientation Mapping (R.Schwarzer)

OIM® - Orientation Imaging Microscopy (TexSEM Laboratories trademark)

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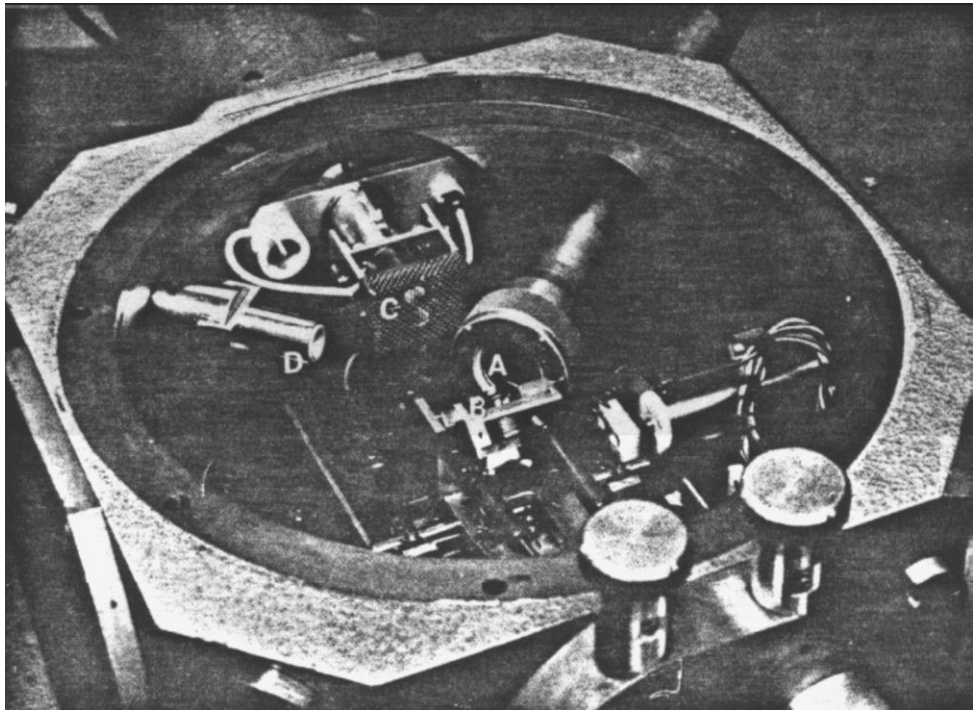
Introduction of the EBSP technique to the SEM

J. A. Venables and C. J. Harland (1973) „Electron Back Scattering Patterns – A New Technique for Obtaining Crystallographic Information in the Scanning Electron Microscope”, *Philosophical Magazine*, **2**, 1193-1200.



Arrangement of the specimen chamber in the Cambridge Stereoscan:
A – Screen, B – Specimen, C – Electron detector, D – X-ray detector

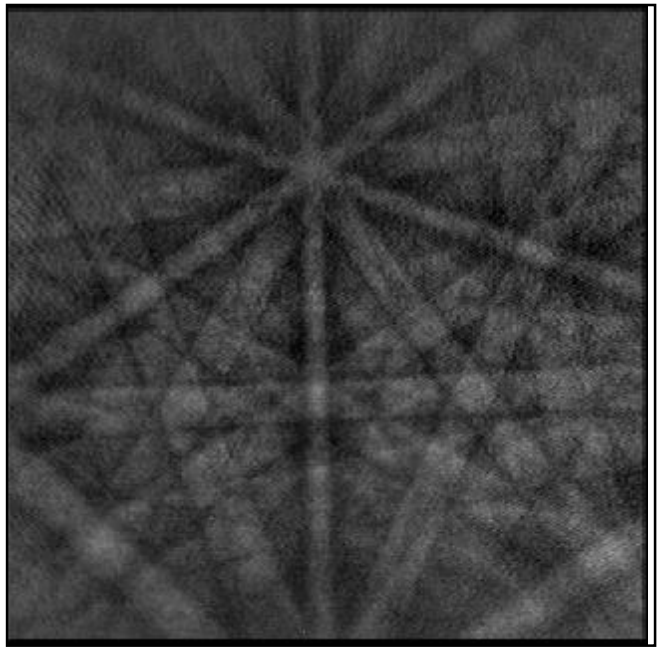
EBSP in the SEM recorded on film

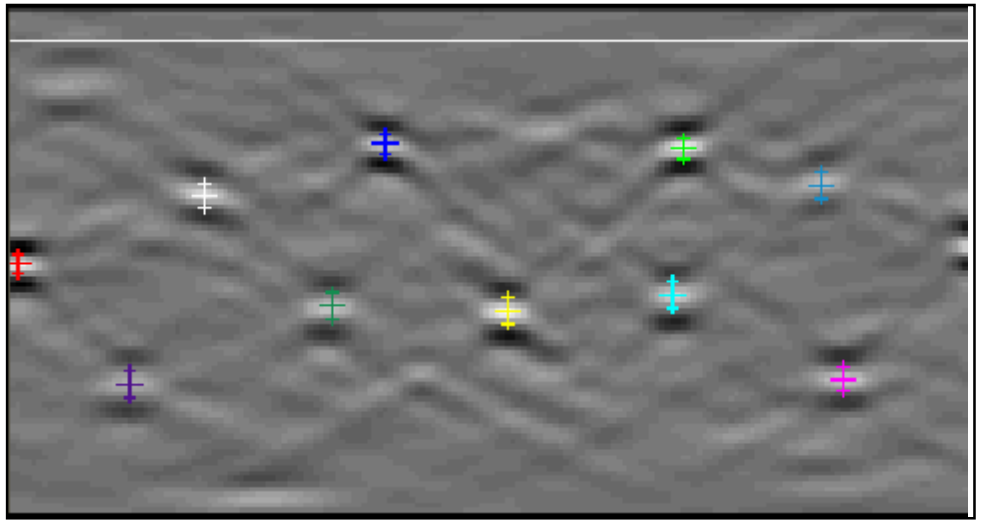
D. J. Dingley (1984) „Diffraction From Sub-Micron Areas Using Electron Backscattering In A Scanning Electron Microscope”, *Scanning Electron Microscopy*, **11**, 569-575

Film replaced by TV camera

D. J. Dingley, M. Longdon, J. Wienbren and J. Alderman (1987) „On-line Analysis of Electron Backscatter Diffraction Patterns, Texture Analysis of Polysilicon”, *Scanning Electron Microscopy* , **11**, 451-456.

N. C. Krieger-Lassen, K. Conradsen and D. Juul-Jensen (1992)
„Image Processing Procedures for Analysis of Electron Back Scattering Patterns”,*Scanning Microscopy,* 6, 115-121.





Hough Transform

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Full automation

K. Kunze, S. I. Wright, B. L. Adams, and D. J. Dingley, 1993 „Advances in Automatic EBSP Single Orientation Measurements”, *Textures Microstructures,* 20, 41-54.

Phase ID

D. J. Dingley and K. Baba-Kishi (1986) „Use of Electron Backscatter Diffraction Patterns for Determination of Crystal Symmetry Elements”, *Scanning Electron Microscopy*, **II**, 383-391.

D. J. Dingley, R. Mackenzie and K. Baba-Kishi (1989) „Application of Backscatter Kikuchi Diffraction for Phase Identification and Crystal Orientation Measurements in Materials”, *Microbeam Analysis*, ed. P.E.Russell, San Francisco Press, 435-436.

J. R. Michael and R. P. Goehner (1993) „Crystallographic Phase Identification in the Scanning Electron Microscope: Backscattered Electron Kikuchi Patterns Imaged with a CCD-Based Detector”, *MSA Bulletin,* **23,** 168-175.

2. EBSD – the basics

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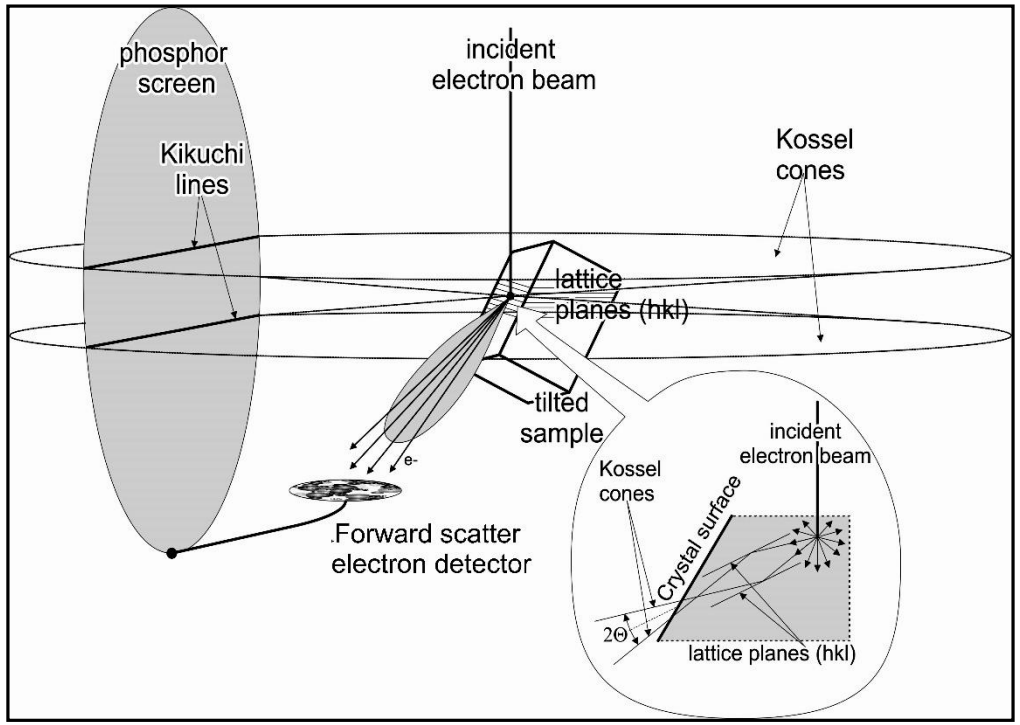
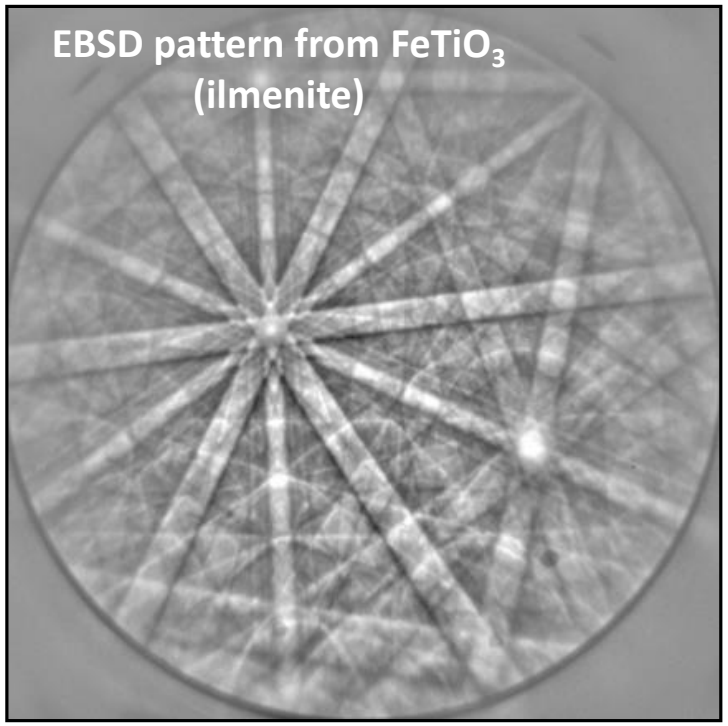
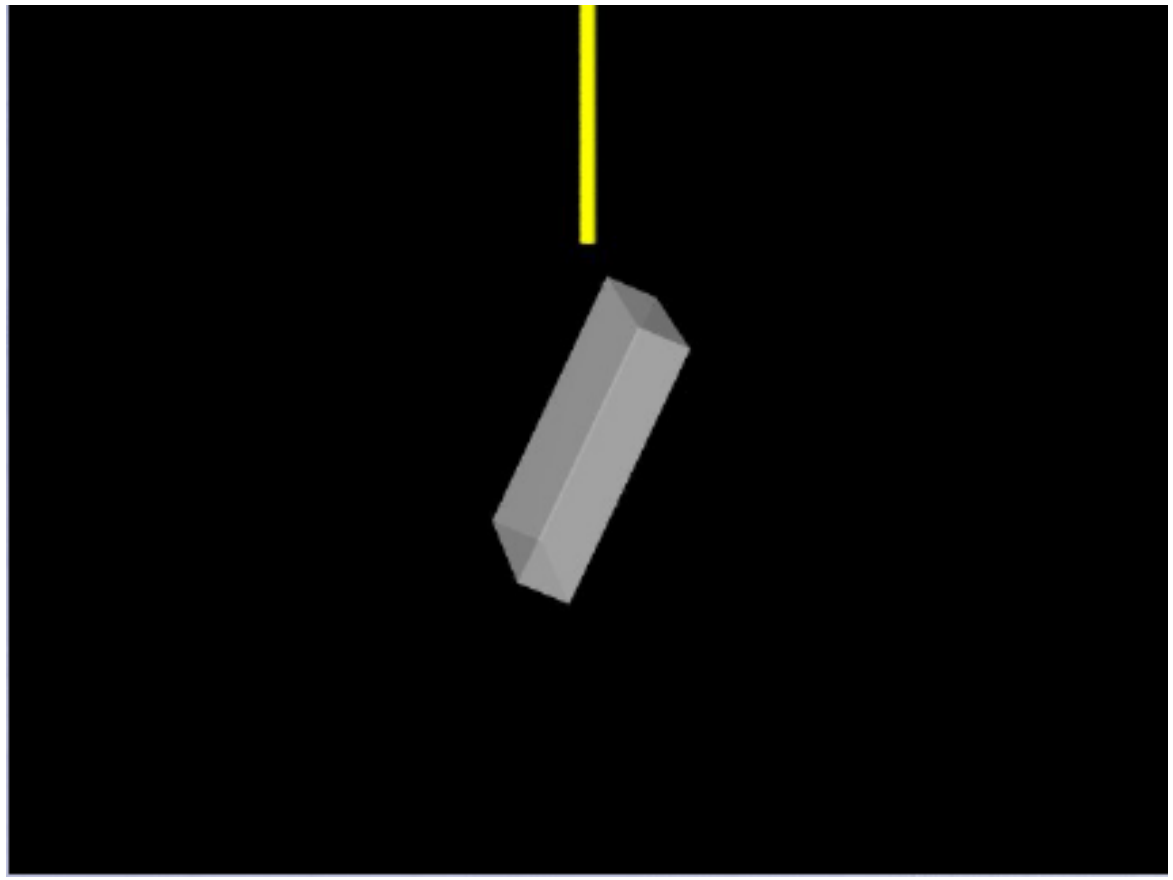
Electron Backscatter Diffraction

- Backscatter electrons interact with the crystal and form for each lattice plane two diffraction cones (so called „Kossel cones" with VERY large conical angles)
- Intersections of diffraction cones with the phosphor screen become visible as a pair of parallel lines – the „Kikuchi bands"







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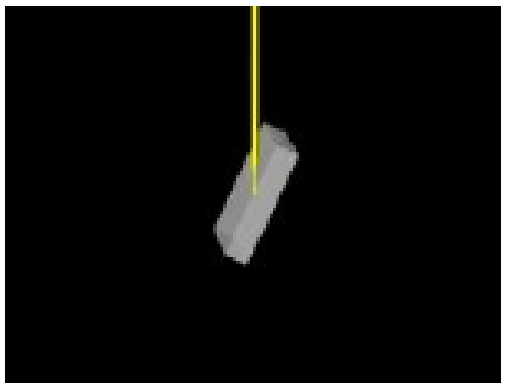

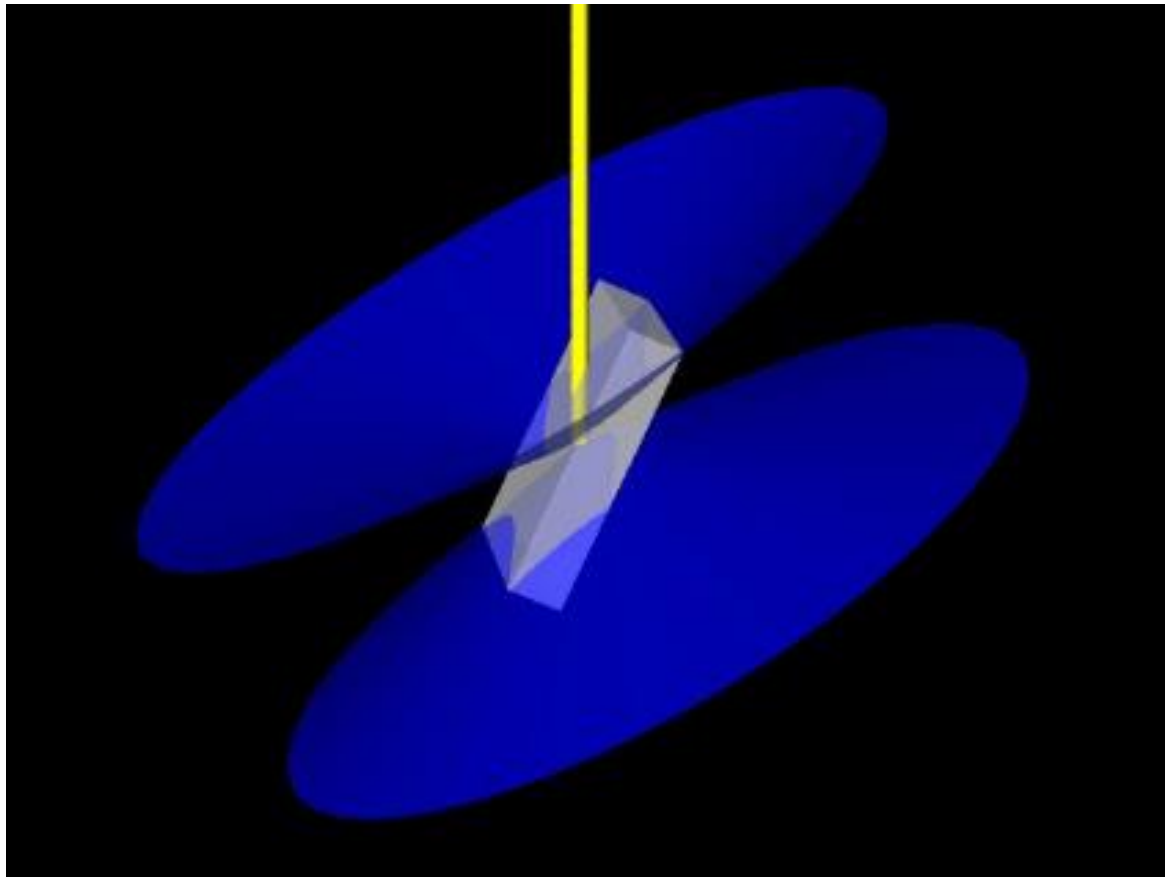

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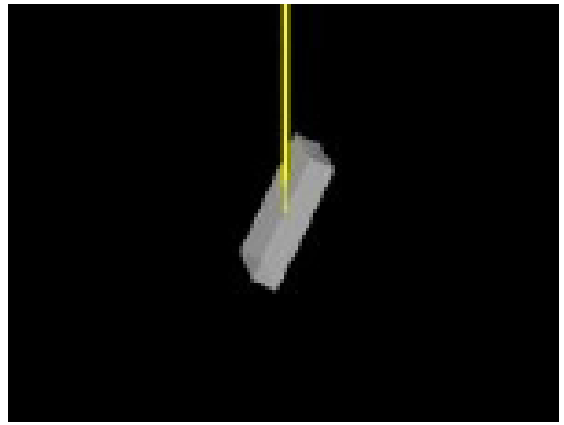

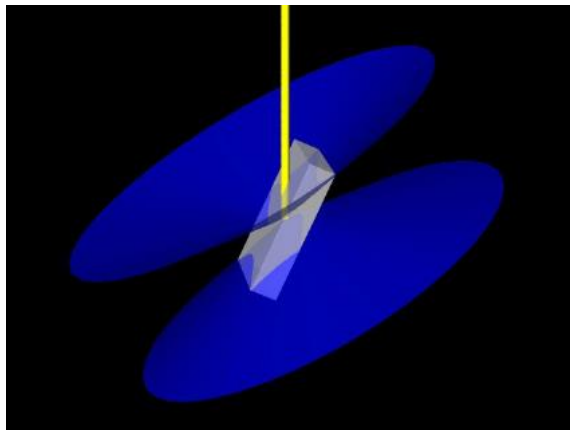

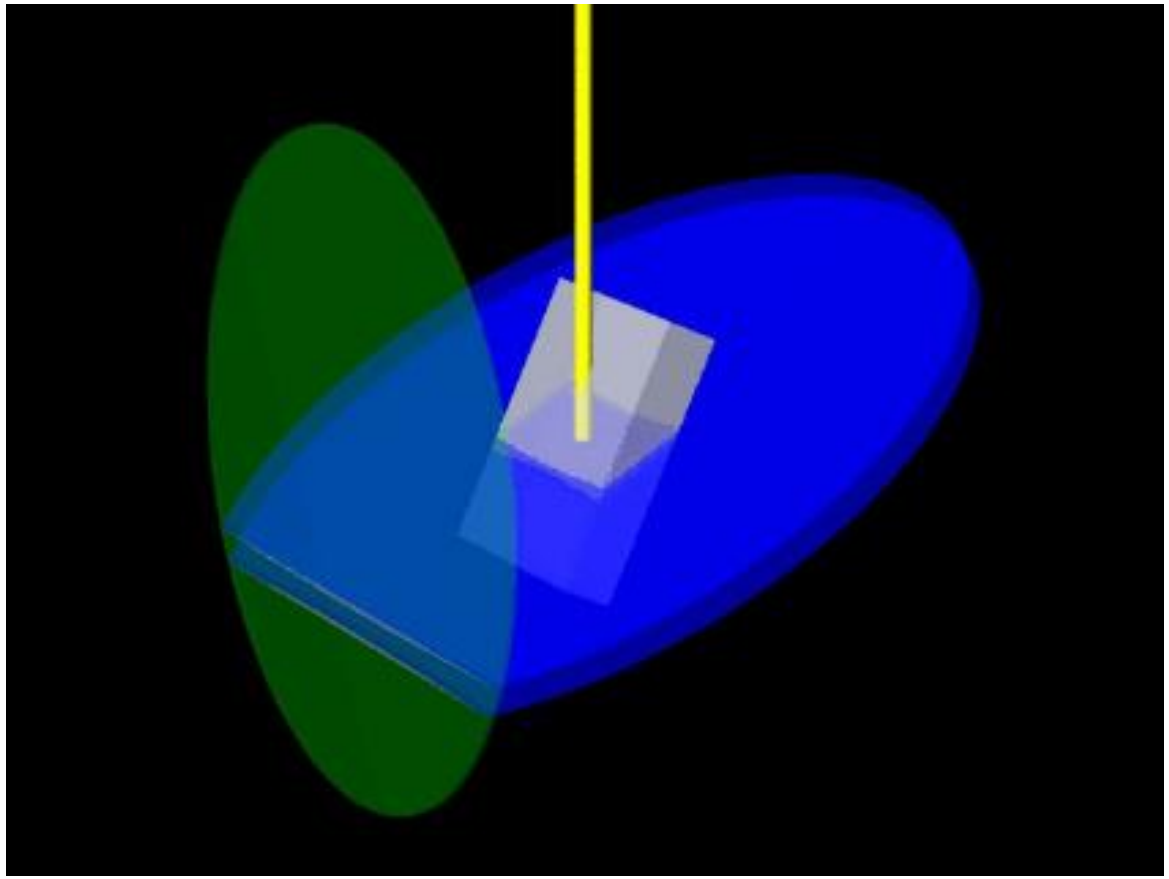

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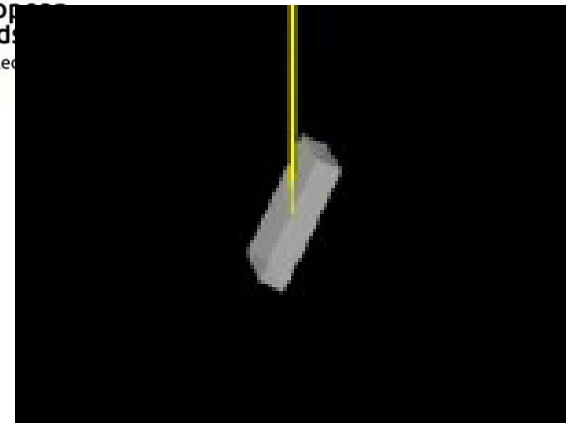

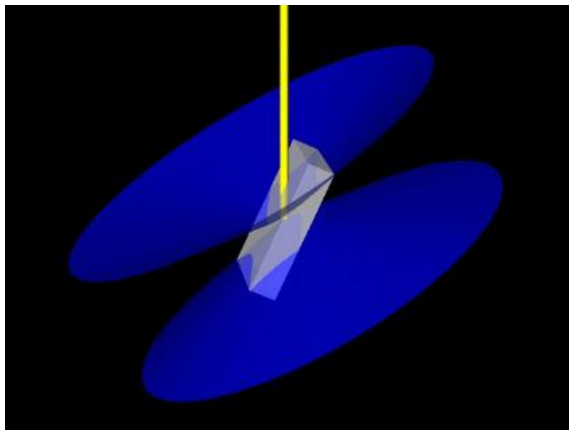

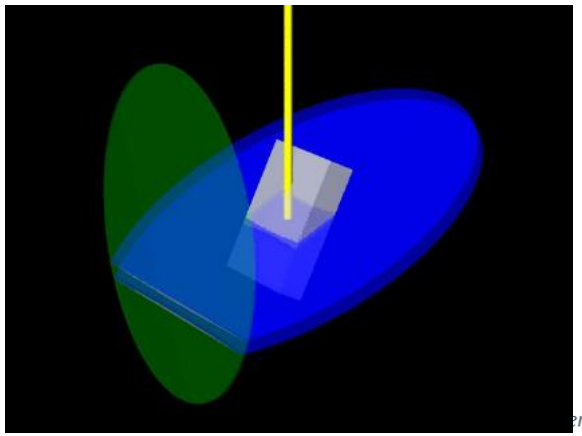

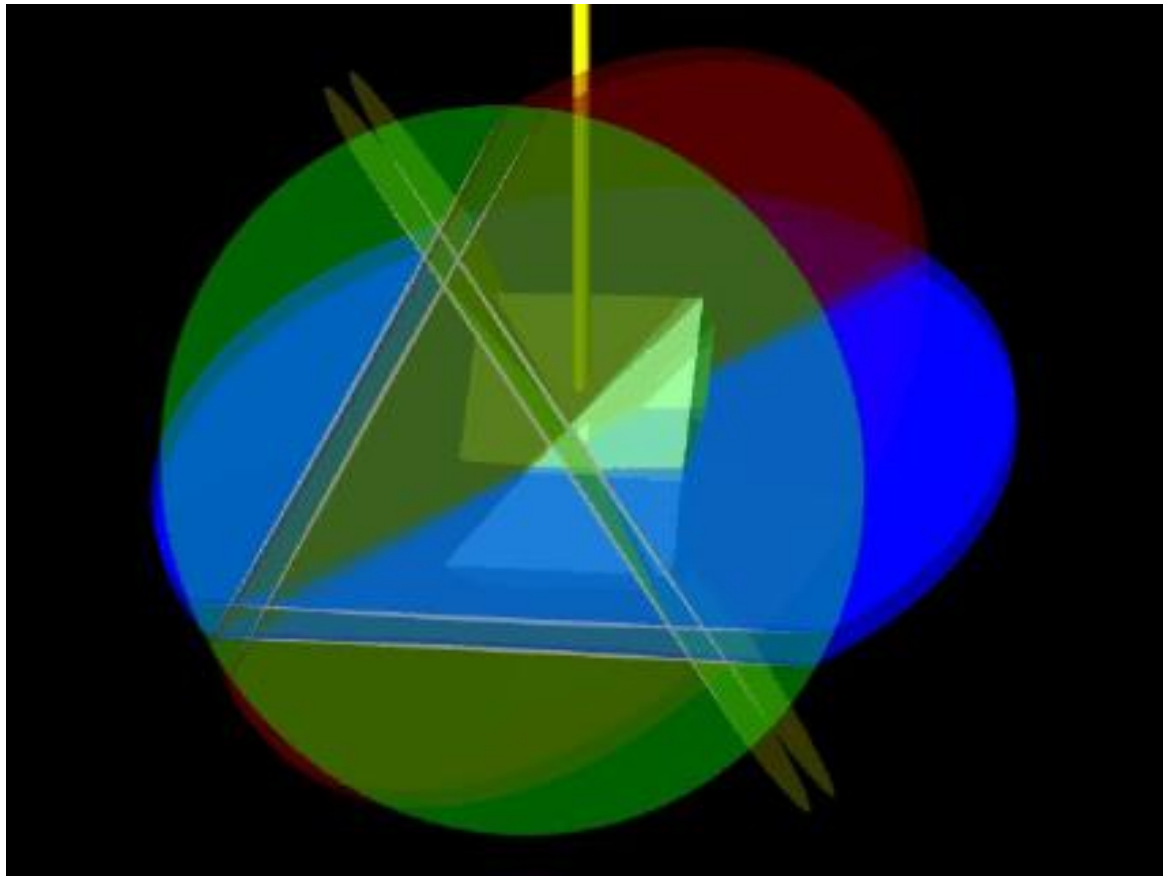

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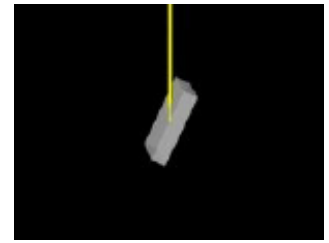

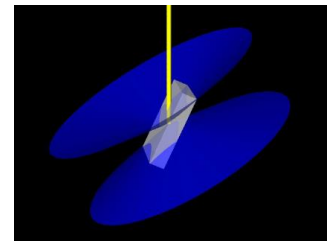

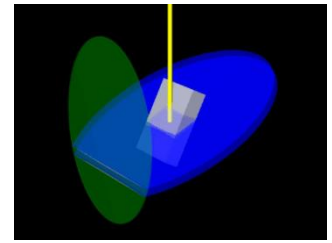

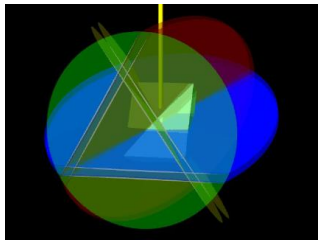

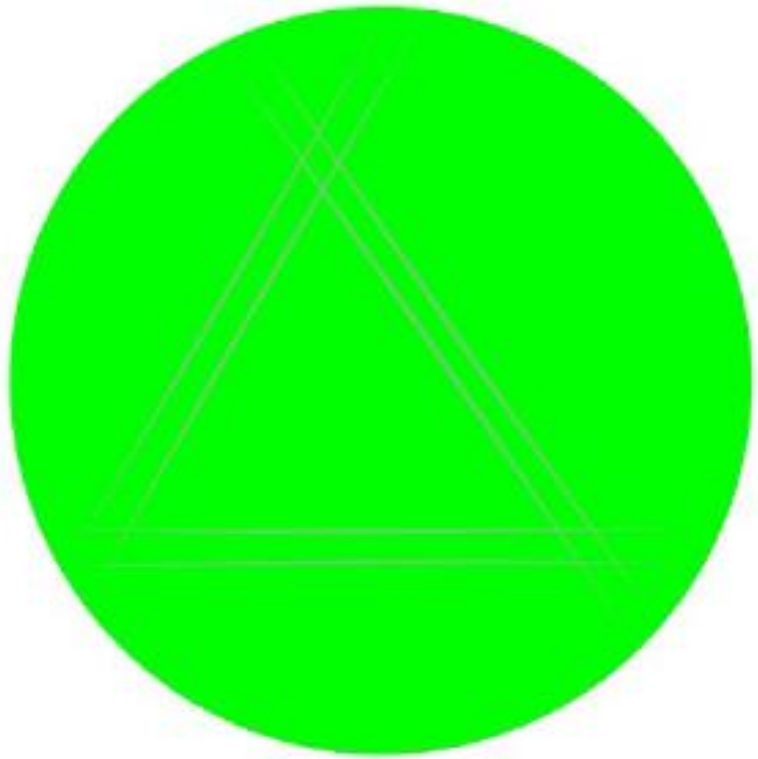

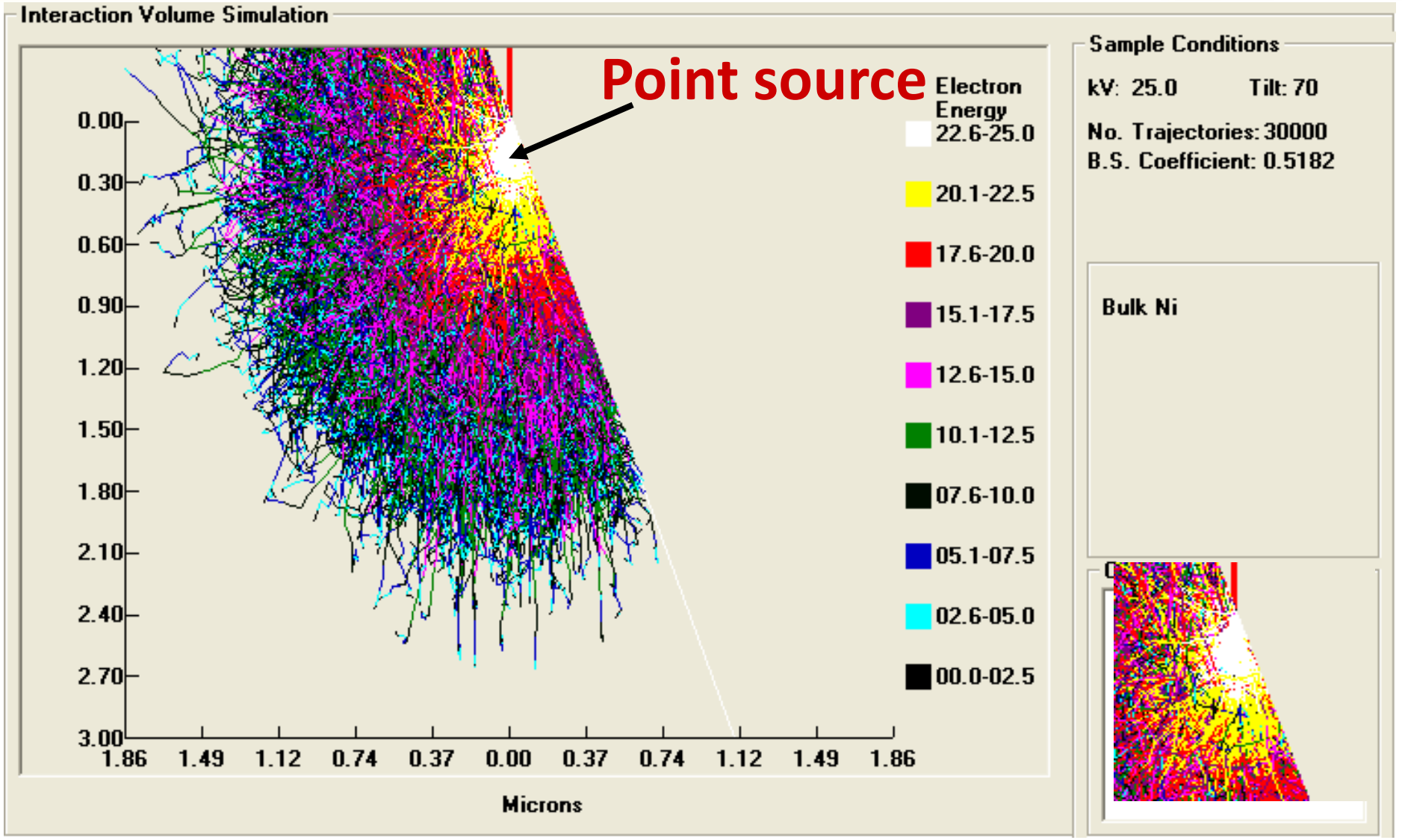

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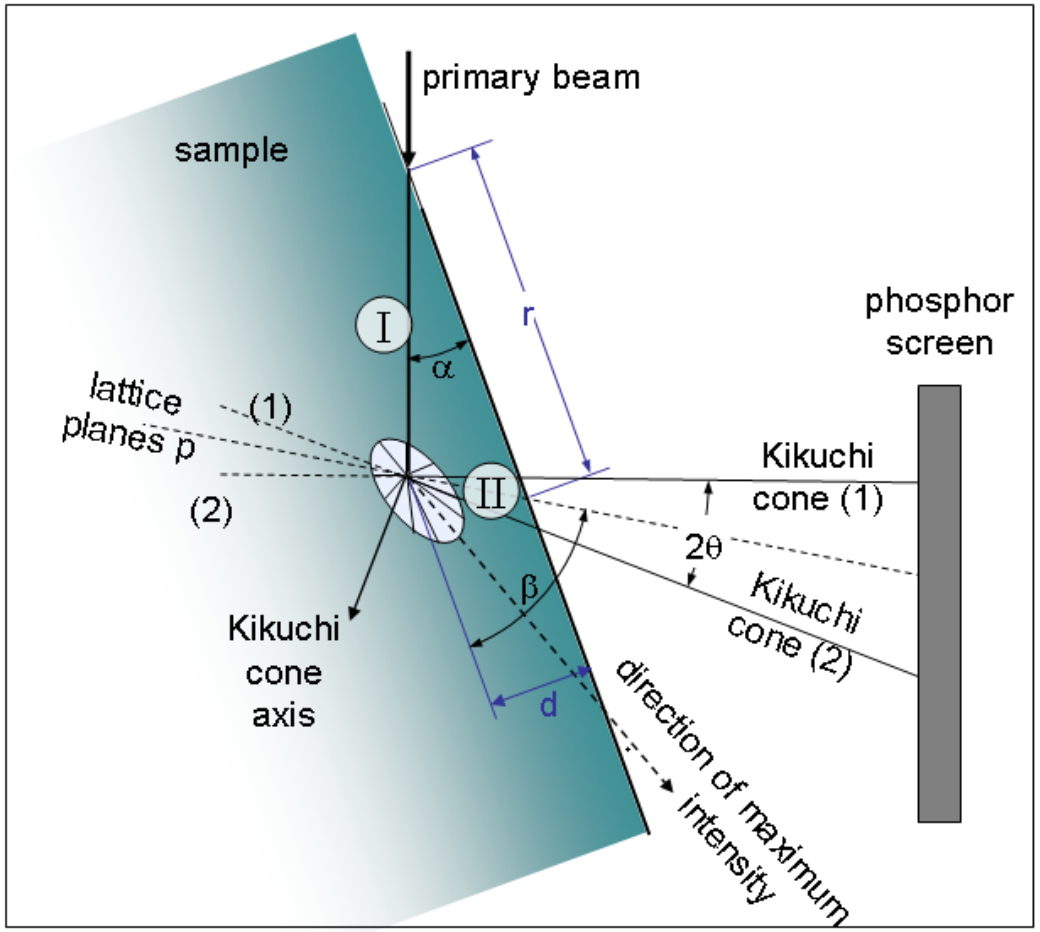
Spatial resolution of EBSD

Step I:
incoherent but quasi-elastic scattering
r: spatial resolution

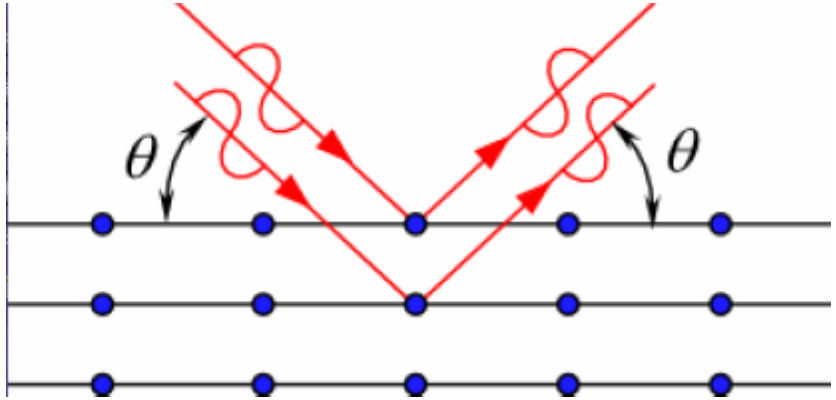
Step II:
coherent scattering from a quasi-point source in the crystal
d: diffraction depth

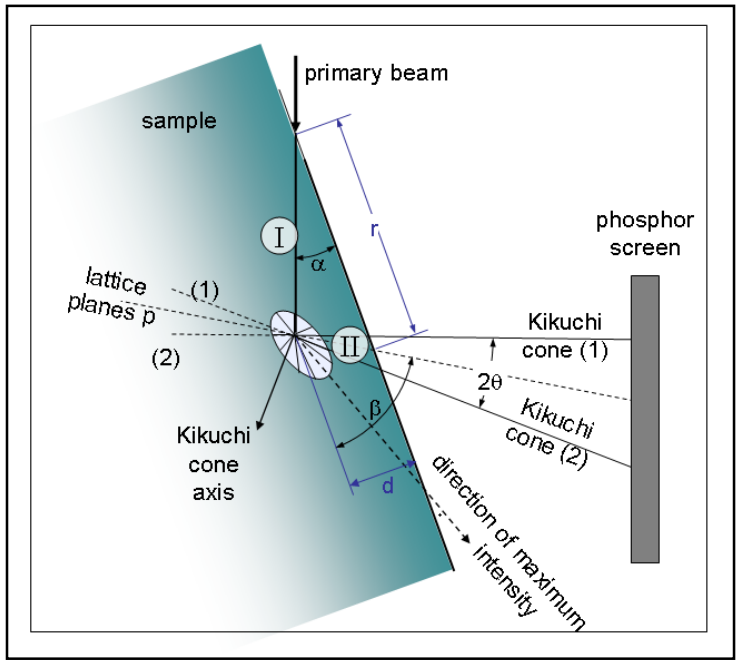


Step 1: electrons enter the material and are quasi-elastically but incoherently scattered. This leads to a wide directional distribution of electrons emerging from a very small volume in the crystal.

<u>This process can be regarded as the formation of an electron source inside the crystal („a point source”).</u> These electrons may now be diffracted according to the Bragg equation.

$$\lambda = 2d_{hkl} \sin\theta$$



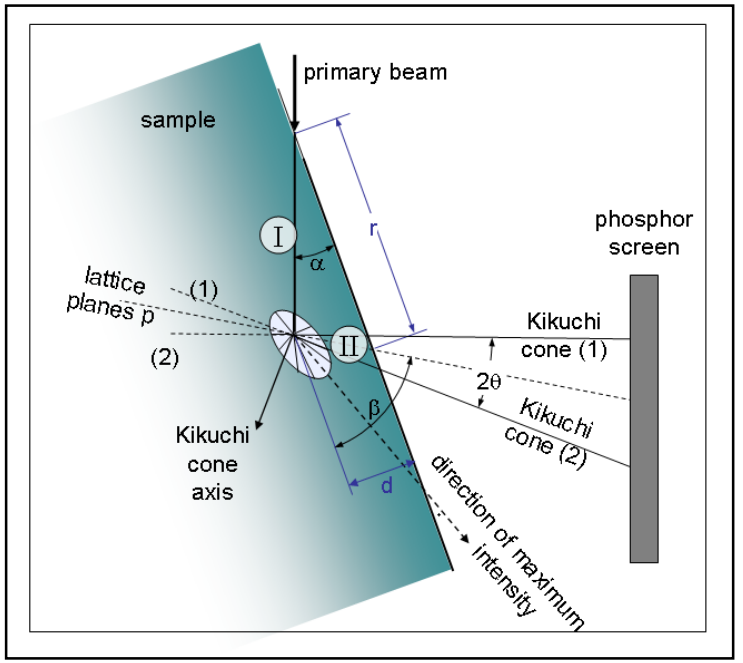


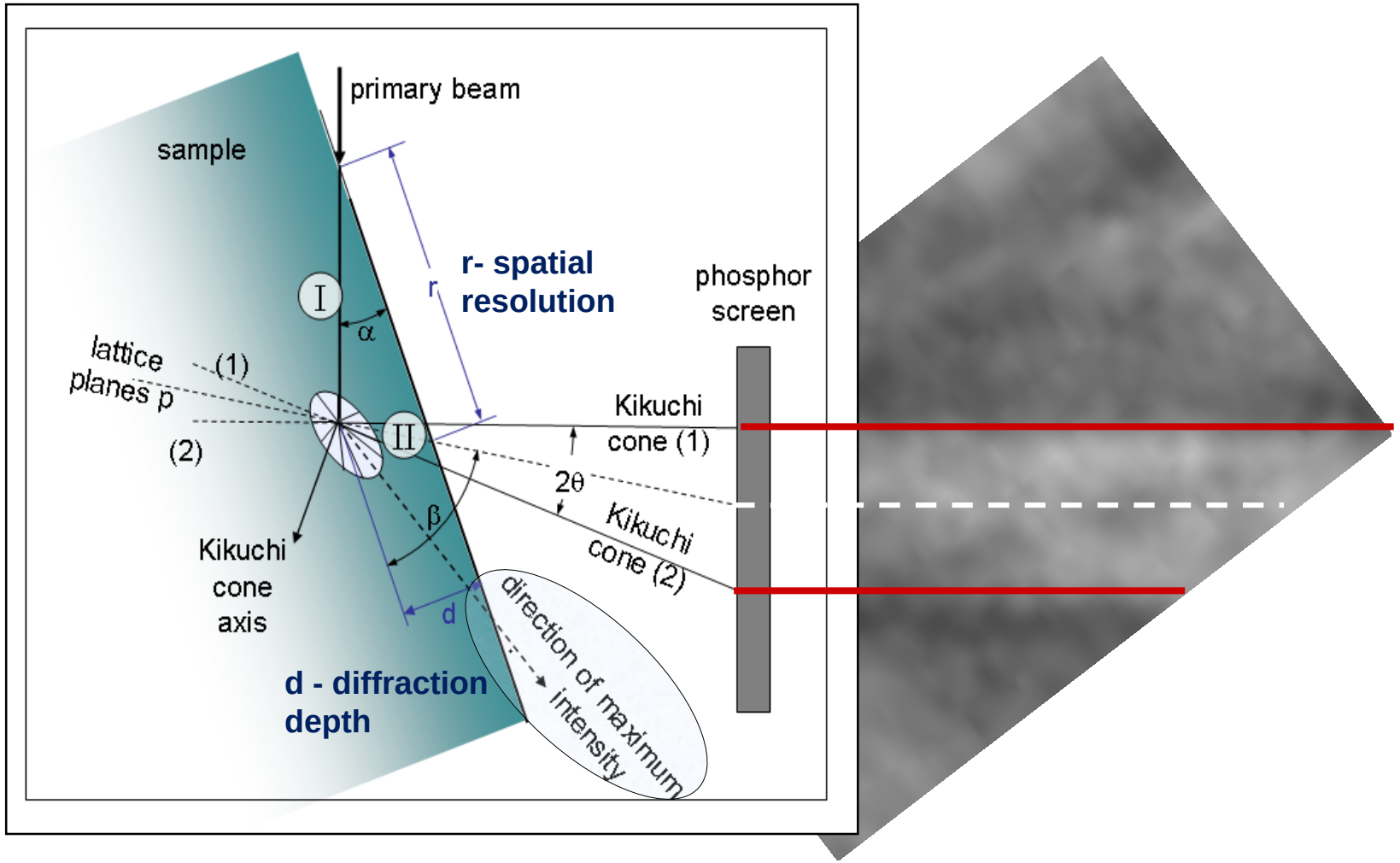
Step 2: diffracted electrons emerging from „the point source" inside of the crystal are coherently scattered (diffracted) by the crystal lattice.

They form **pairs of cones centred about the lattice plane normal vectors with the operating angle 180^o - 2θ and the angle of 2θ between them**.

Since the Bragg angle is rather small (in order of 1^o for 15 kV accelerating voltage) – the cones are very large. Each pair of cones intersects phosphor screen in almost straight and parallel lines.

The distance between the two lines is approximately proportional to tan(θ), while the position of the centre line can be understood as the **gnomonic projection** of the diffraction plane onto the observation screen.





A very surface sensitive technique

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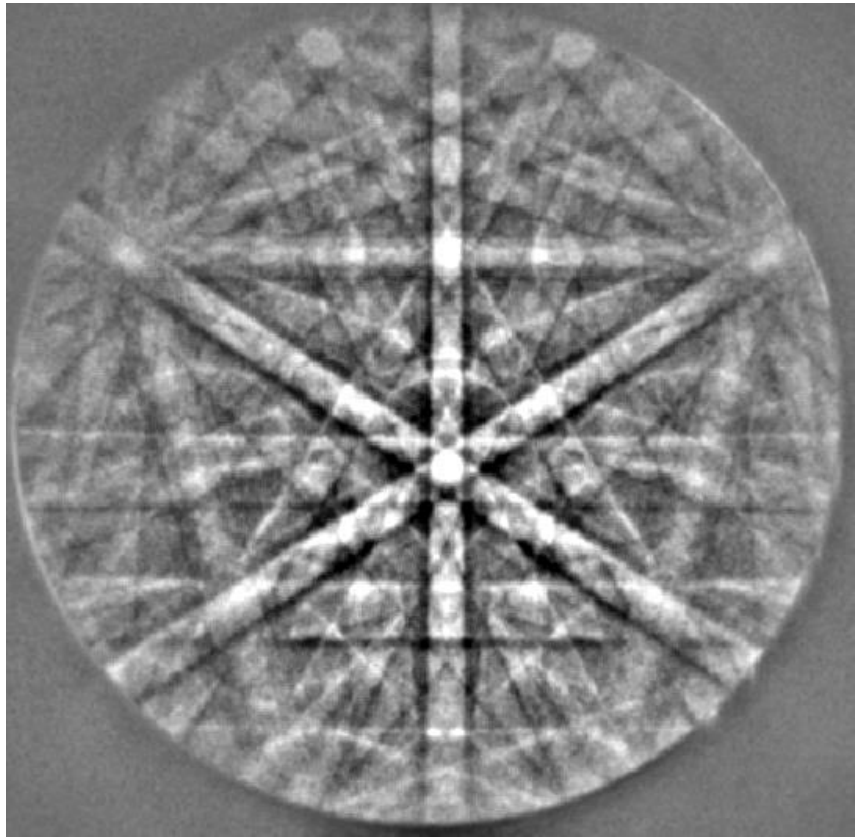
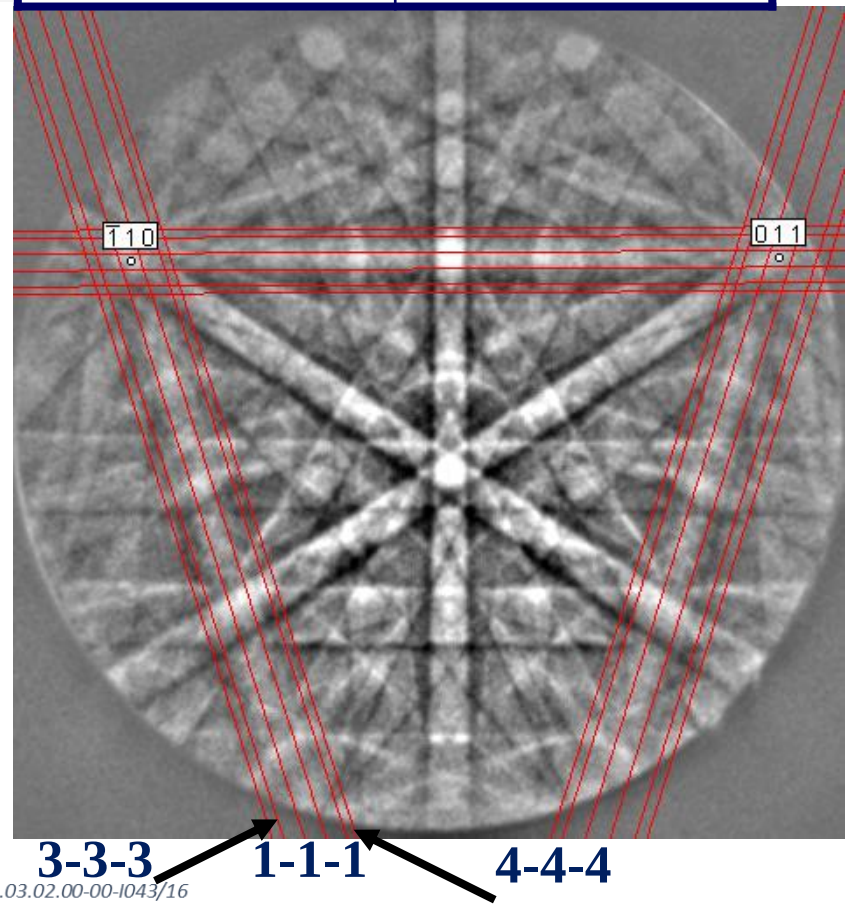
Band width is inversely proportional to d-spacing

Si 1-1-1	3,135 A
Si 3-3-3	1,045 A
Si 4-4-4	0,784 A





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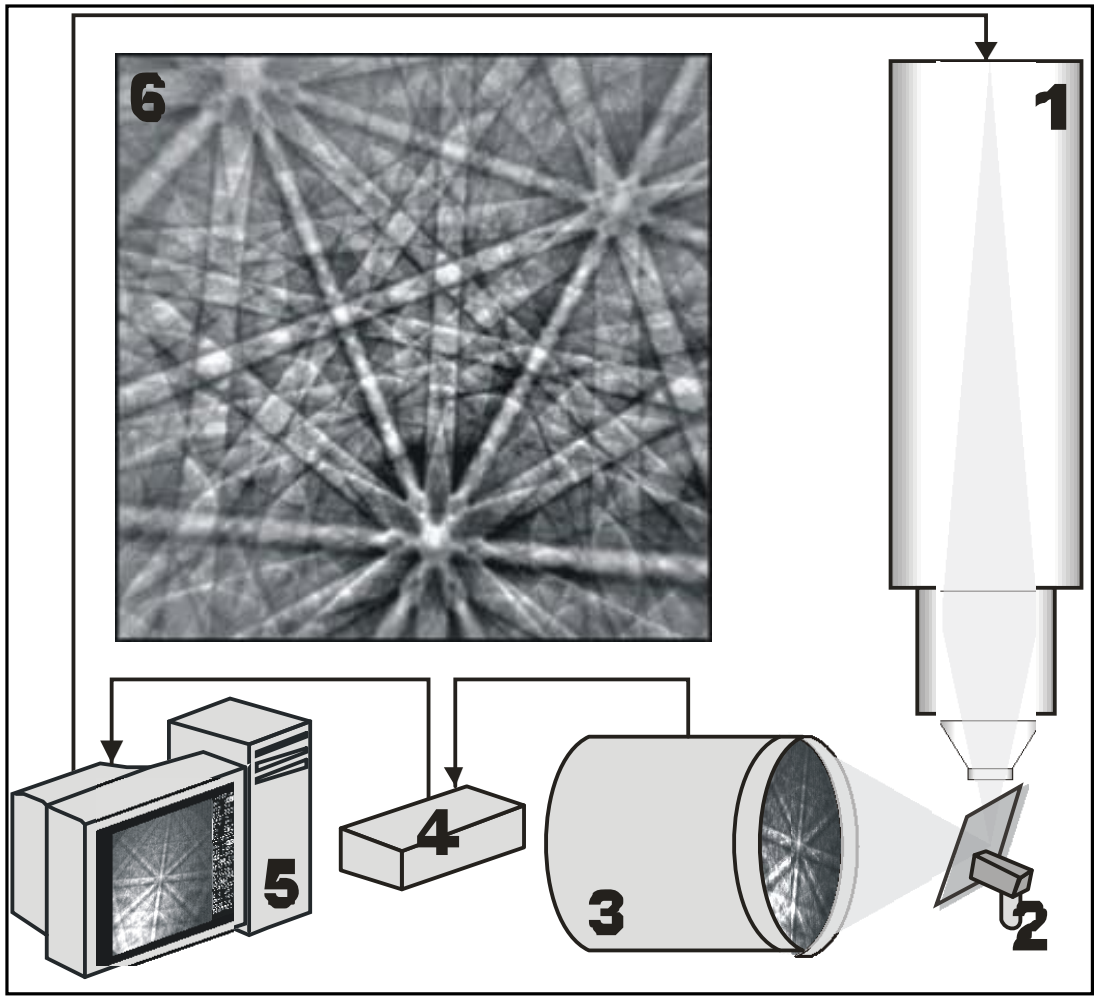
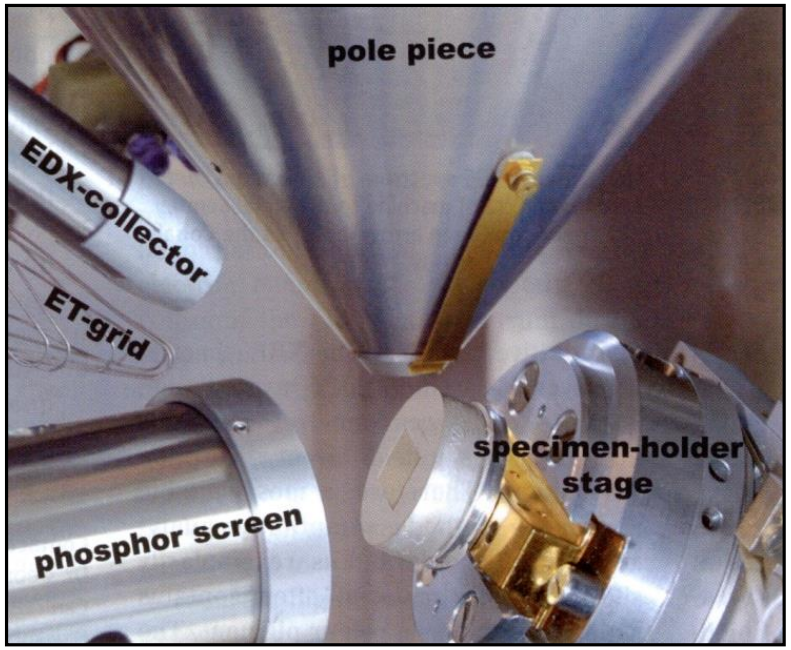


1. SEM
2. A sample tilted at 70° from the horizontal
3. A phosphor screen which is fluoresced by electrons from the sample to form the diffraction pattern. A sensitive charge coupled device (CCD) video camera for viewing the diffraction pattern on the phosphor screen image processor
4. Image processor (at present embedded in the CCD camera)
5. A computer to control EBSD experiments, analyze the diffraction pattern and process and display the results
6. Diffraction pattern



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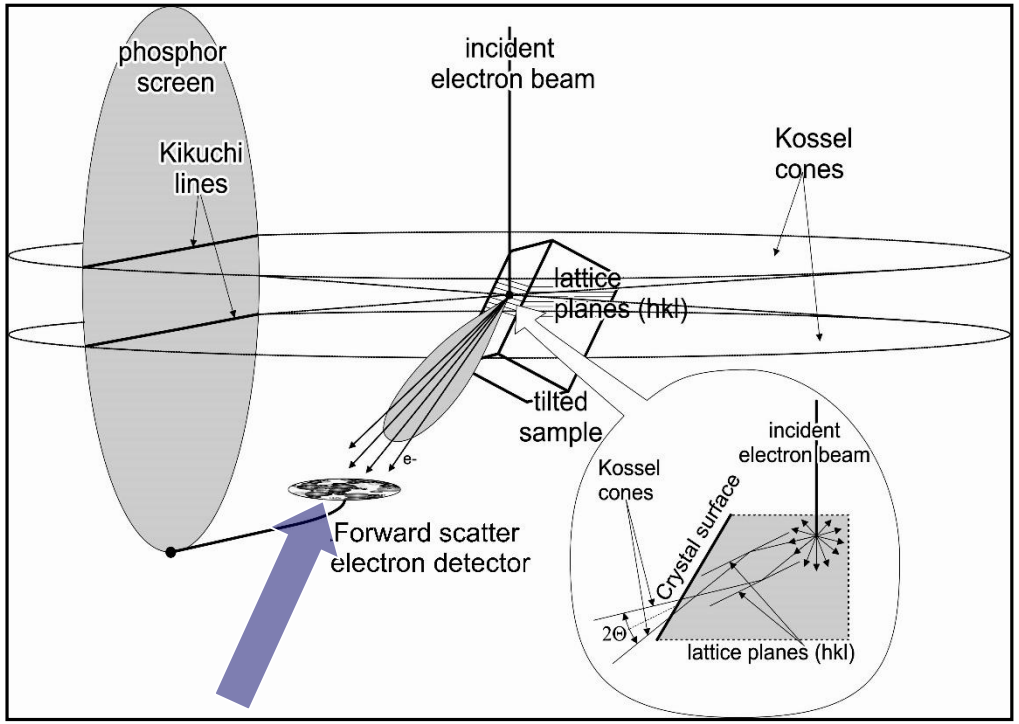
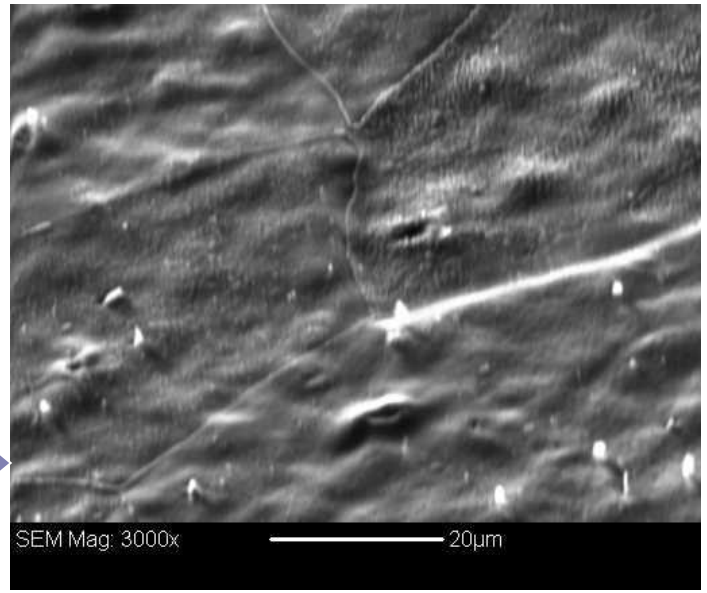
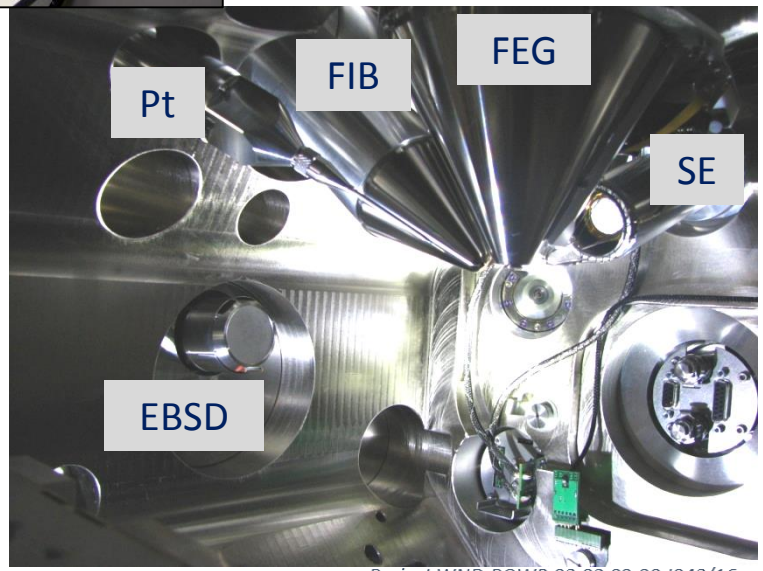


An optional electron solid state detector mounted below the phosphor screen for electrons scattered in the forward direction from the sample

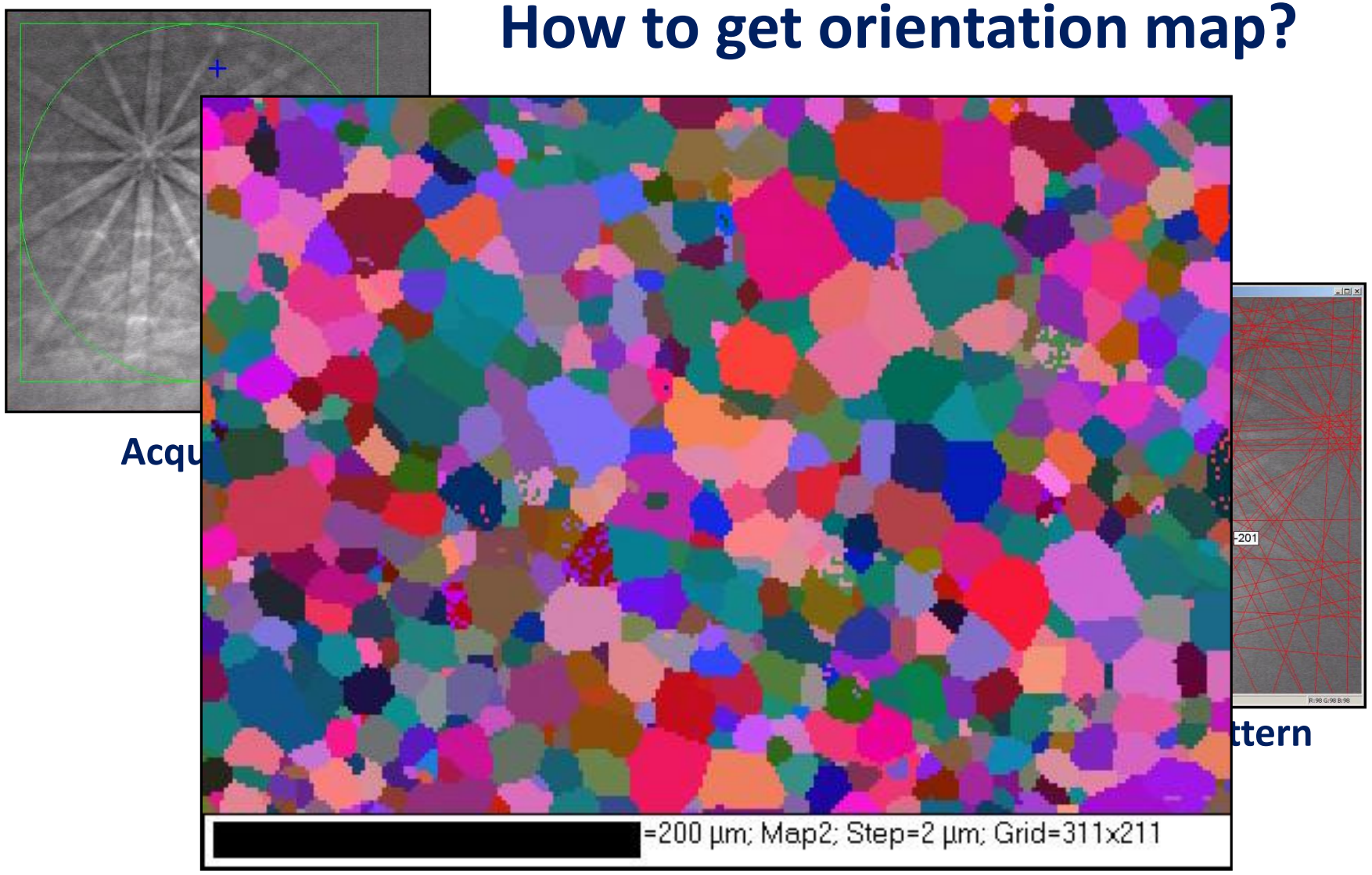


➢**Post-processing of diffraction data can produce a lot of additional information like lattice strain, size, shape and type of boundaries of grains etc.**

➢**The results are „comparable” with X-ray diffraction experiments but have advantage of the local assignment!**



How to get orientation map?



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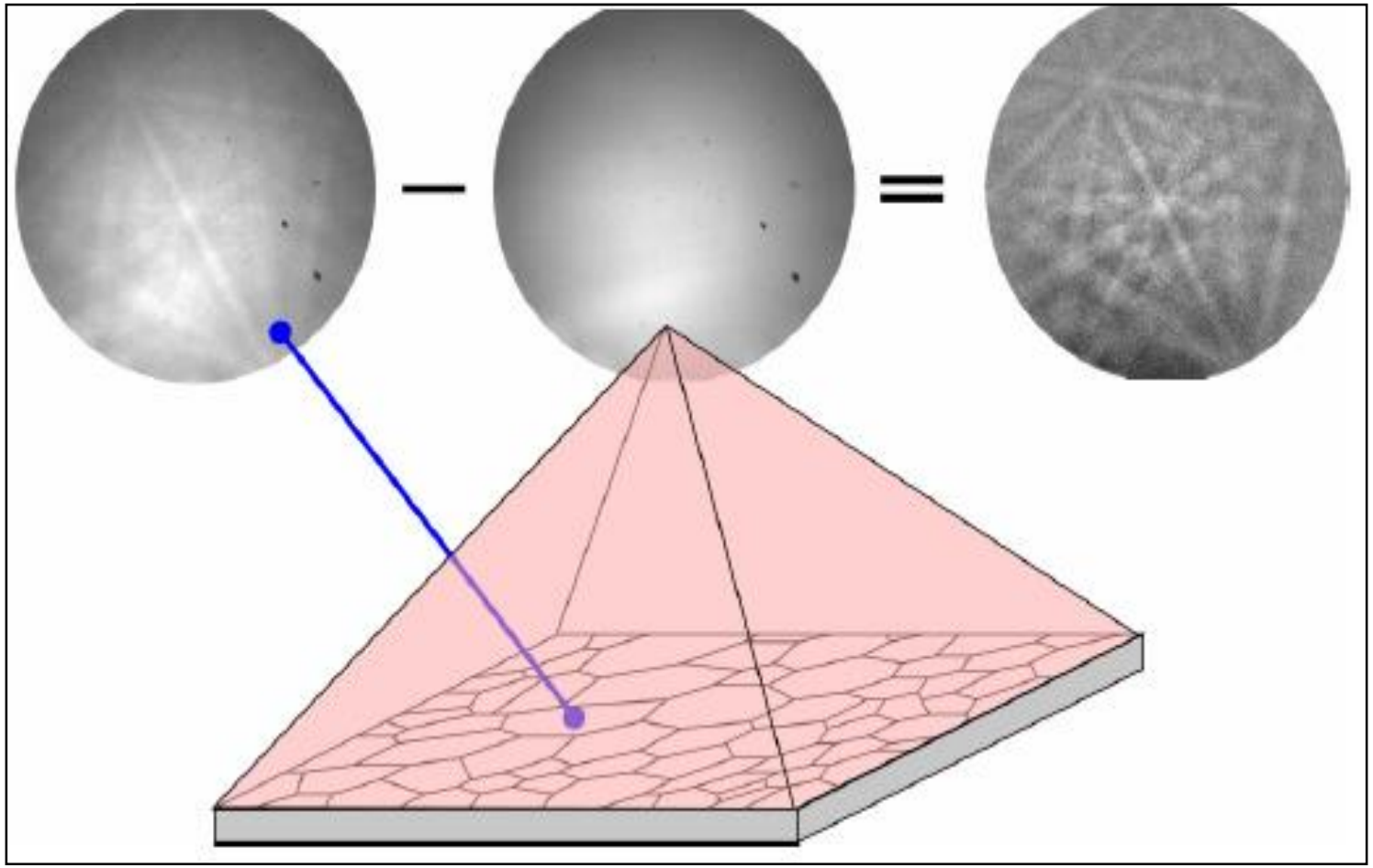
Acquisition – Background subtraction



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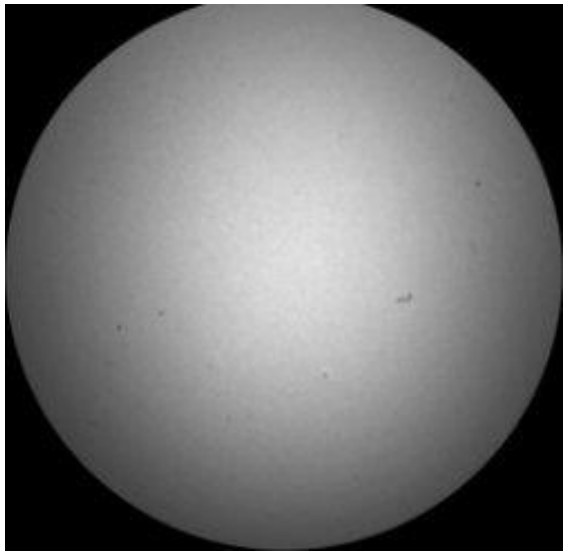
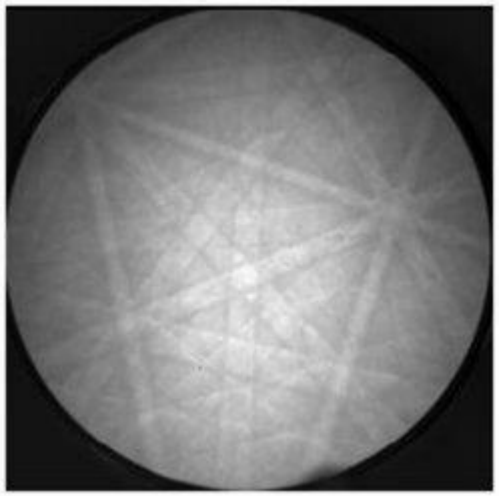
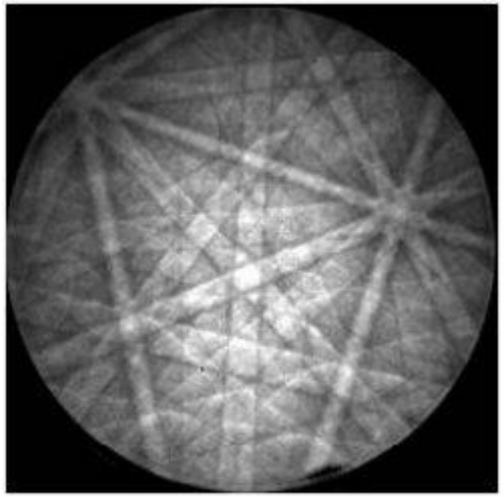
Background



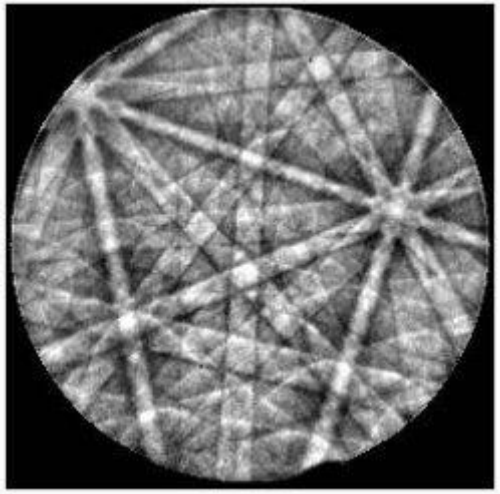
Original pattern



Background subtraction



Background division



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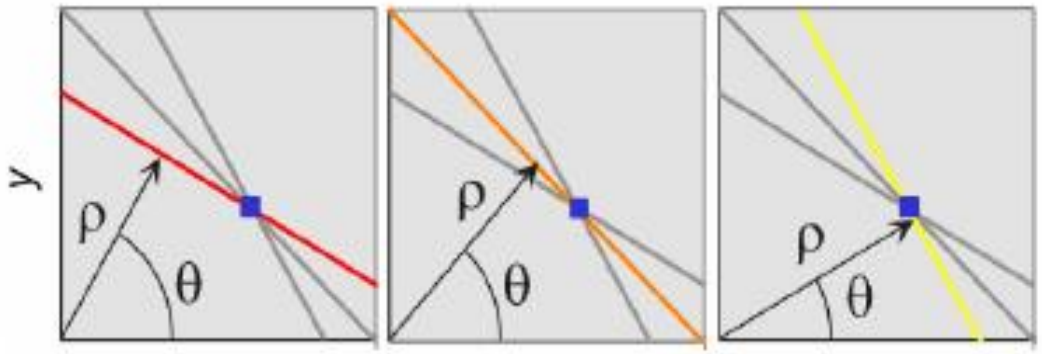
Detection - Hough Transform

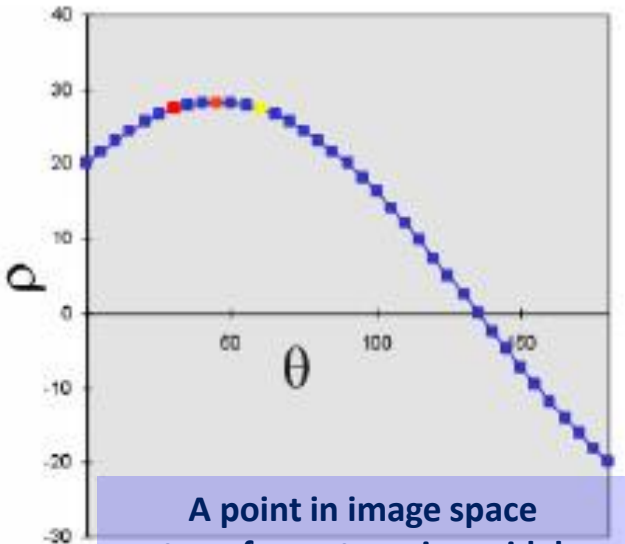
P.C.V. Hough „Method and means for recognising complex pattern”

US Patent 3 069 654, 1962

- A given pixel in an image can belong to an infinitive number of lines.
- A line can be parameterized by the Hough parameters „ρ” and „θ”.
- ρ represents the perpendicular distance of the line of the origin,
- θ describes the angle of the line.
- The relationship between the lines passing through a pixel at a coordinate in the image of x and y can be expressed as:

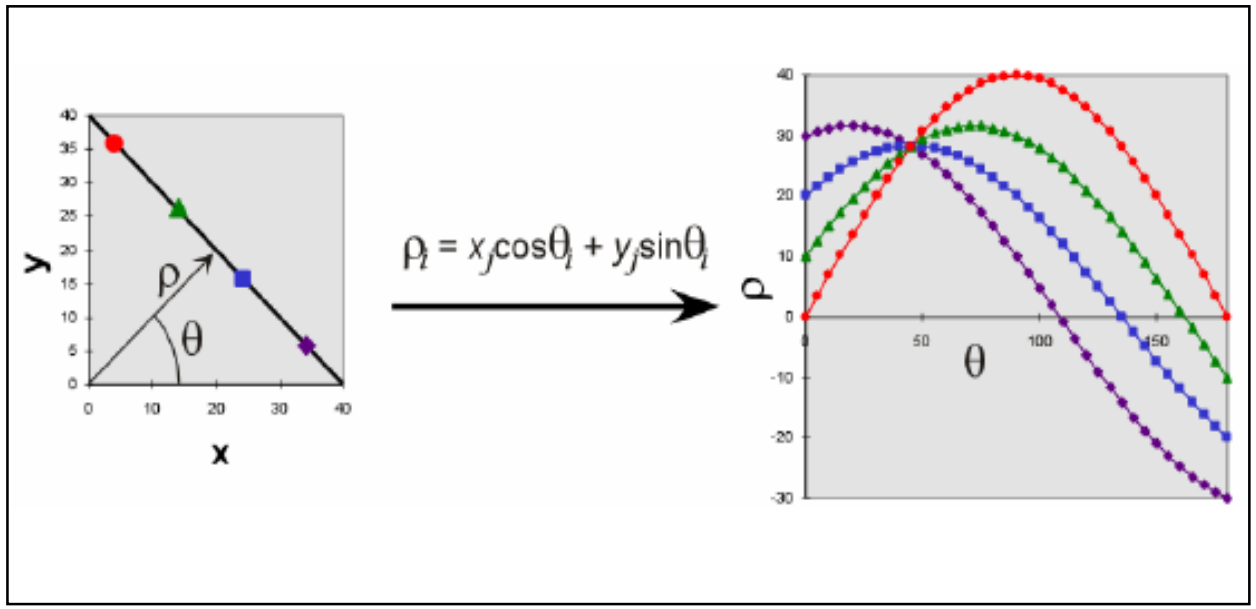
$$\rho_i = x\cos\theta_i + y\sin\theta_i$$

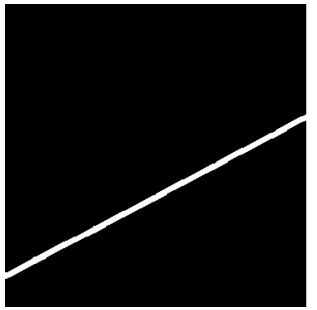


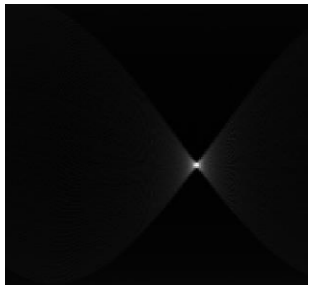


A point in image space transforms to a sinusoidal curve in Hough space!

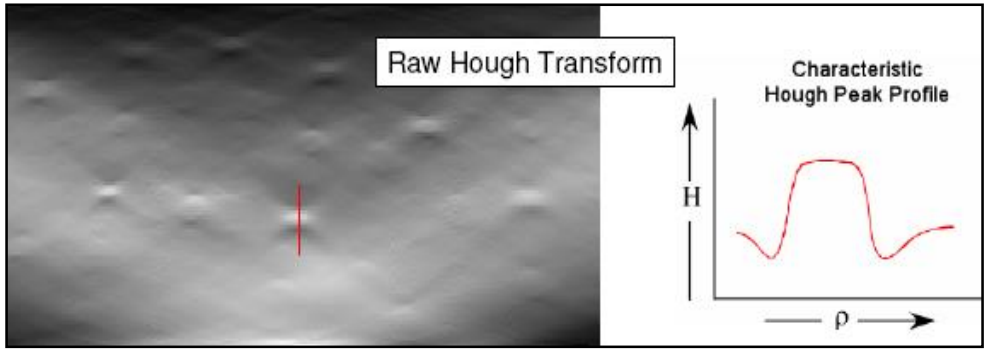
- Consider 4 pixels along a line.
- For each pixel in the line, all possible ρ values are calculated for θ ranging in values from 0 to 180° using the equation: $\rho = x\cos\theta + y\sin\theta$. This produces 4 sinusoidal curves.
- These curves intersect at a point at „ρ" and „θ" coordinates corresponding to the angle of the line (θ) and its position relative to the origin (ρ).
- Thus, a line in image space transforms to a point in Hough Space.

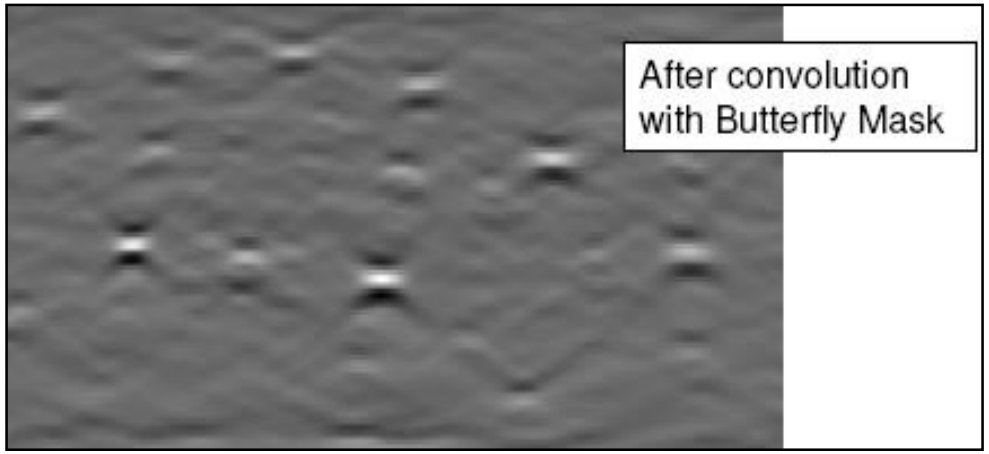






Detection - Hough transform





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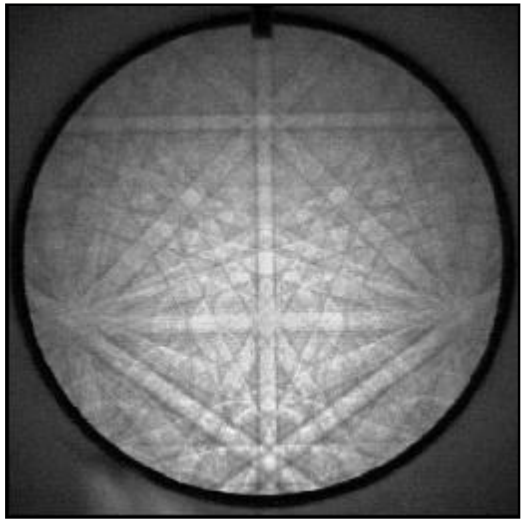

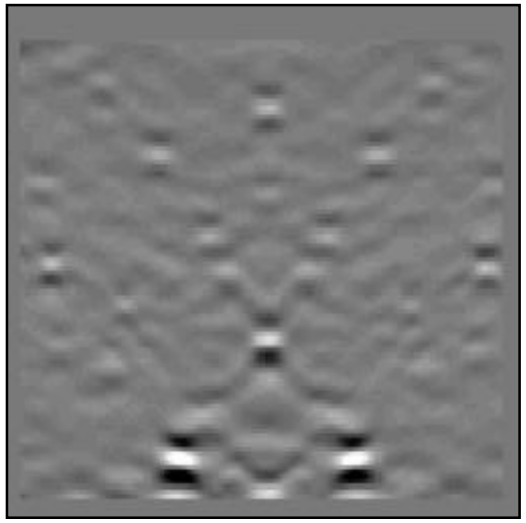

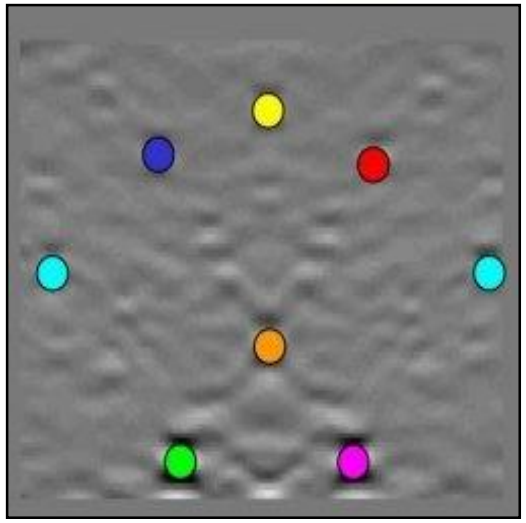

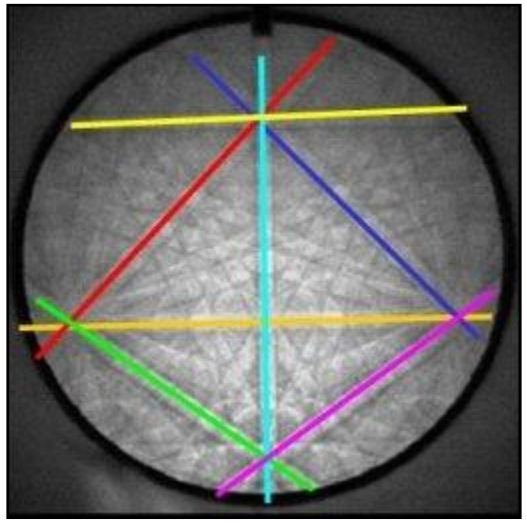

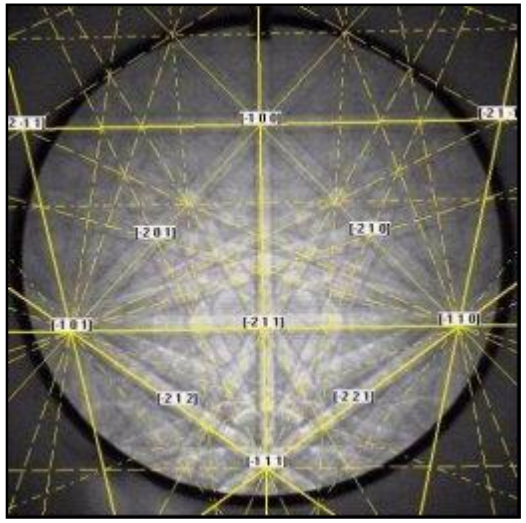

Detection
Hough transform

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Structure factor

For n number of atoms in unit cell:

$$F_{hkl} = \sum_{j=1}^{n} f_j(\theta) \exp[-2\pi i(hu_j + kv_j + lw_j)]$$

In some case crystallographic planes do not diffract electrons – diffraction does not occur – *forbidden reflections*

for f.c.c. (Au) – Au atoms on the corners of unit cell and in the middle of unit cell planes.
4 atoms at the following position [0,0,0], [0,0.5,0.5], [0.5,0,0.5], [0.5,0,5,0]

Structure factor

$$F_{hkl} = f_1(\theta)\begin{pmatrix} \exp[-2\pi i(1\cdot 0+0\cdot 0+0\cdot 0)] \\ +\exp[-2\pi i(1\cdot 0+0\cdot \frac{1}{2}+0\cdot \frac{1}{2})] \\ +\exp[-2\pi i(1\cdot \frac{1}{2}+0\cdot 0+0\cdot \frac{1}{2})] \\ +\exp[-2\pi i(1\cdot \frac{1}{2}+0\cdot \frac{1}{2}+0\cdot 0)] \end{pmatrix} = \begin{pmatrix} \exp[\cdot 0)] \\ +\exp[0] \\ +\exp[-\pi i] \\ +\exp[-\pi i] \end{pmatrix} = 0$$

For practical reasons „a developed formula" for F_{hkl} calculations is used:

$$F_{hkl} = \sum_{j=1}^{n} f_i(\Theta)\cos 2\pi(hx_n + ky_n + lz_n) - i\sum_{j=1}^{n} f_i(\Theta)\sin 2\pi(hx_n + ky_n + lz_n)$$

x, y, z **– position of „n" atom**

hkl **– Miller indices**

http://www.ftj.agh.edu.pl/~Wierzbanowski/Dyfrakcja.pdf

Al

Reflectors	No.	d-spacing, Å	Intensity %
{111}	4	2.338	100.0
{200}	3	2.025	69.4
{202}	6	1.432	27.6
{113}	12	1.221	18.2
{222}	4	1.169	16.2
{400}	3	1.013	11.2
{331}	12	0.929	9.0
{402}	12	0.906	8.4
{422}	12	0.827	6.6
etc.			

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Mechanisms giving rise to the Kikuchi band intensities and profile shapes are complex. As an approximation, the intensity of a Kikuchi band for the plane (hkl), on which diffraction takes place, is given by:

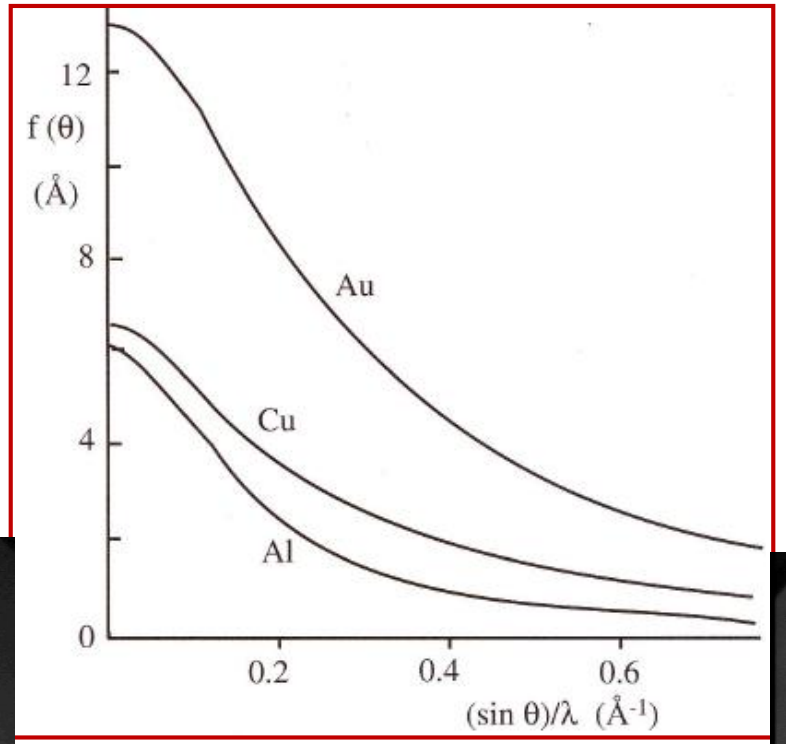
$$I_{hkl} = \left[\sum_i f_i(\Theta)\cos 2\pi(hx_i + ky_i + lz_i)\right]^2 + \left[\sum_i f_i(\Theta)\sin 2\pi(hx_i + ky_i + lz_i)\right]^2$$

$$\mathbf{I_{hkl} = \sum f_i(\Theta)\left[\cos 2\pi(hx_i + ky_i + lz_i)^2 + \sin 2\pi(hx_i + ky_i + lz_i)^2\right]}$$

where: $f_i(\theta)$ is the atomic scattering factor for electrons and (x_i y_i z_i) are the fractional coordinates in the unit cell for atom *i* (atom positions).

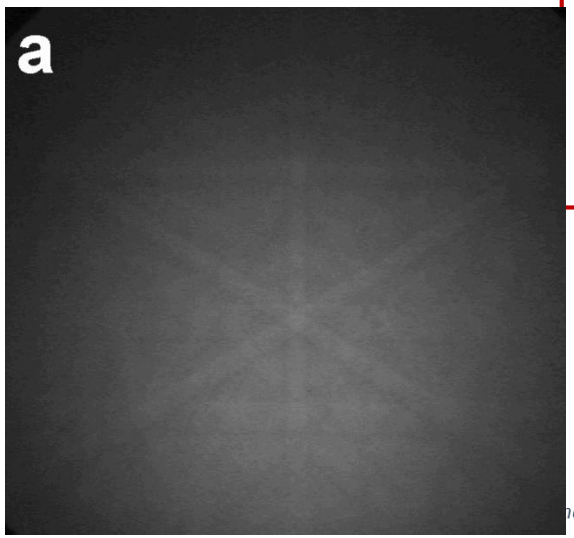
An observed diffraction pattern should be compared with a calculated simulation using above equation to ensure only planes that produce visible Kikuchi bands are used when solving the diffraction pattern. This is especially important when working with materials with more than one atom type.

$$f(\theta) = \frac{m_o e^2}{2h^2}\left(\frac{\lambda}{\sin\theta}\right)^2 (Z - f_x)$$

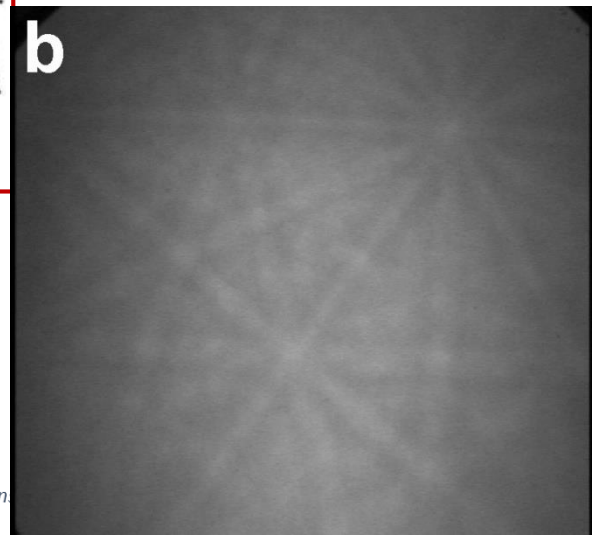


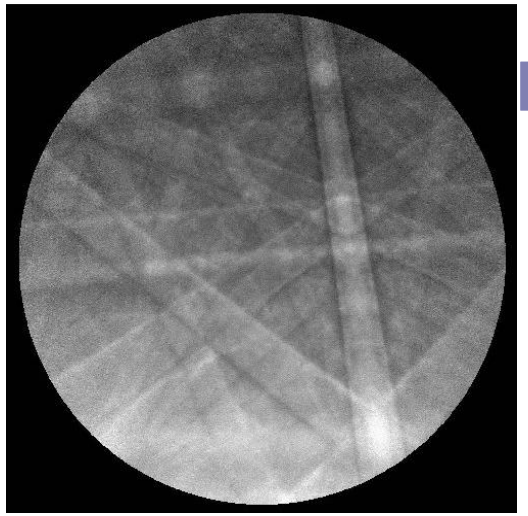
where:
Z – atomic number
f_x – scattering factor for x-rays
$(\lambda/\sin\theta)$ – Rutherford scattering of electron on atomic core
$(Z–f_x)$ – scattering of electron on inner shell electrons
(L.REIMER)

Al

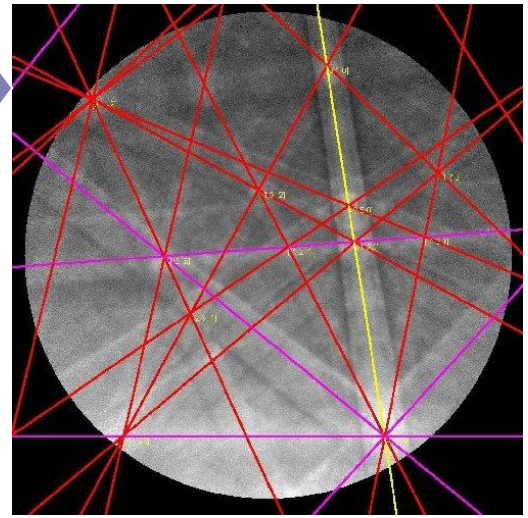


Ta

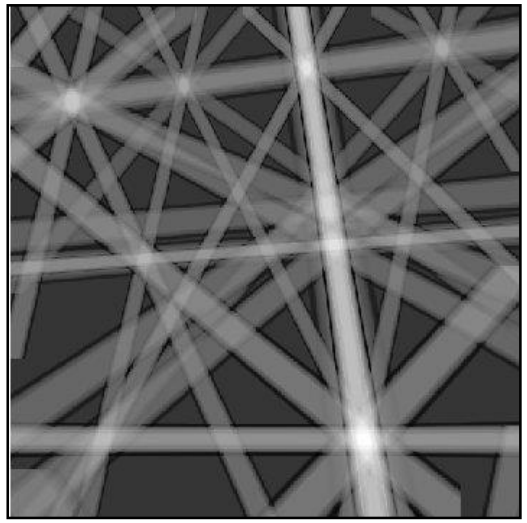




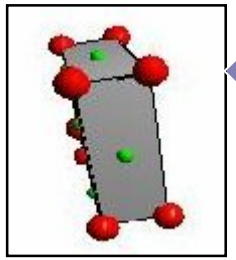
Diffraction pattern from the orthorhombic ceramic mullite ($3Al_2O_3$ $2SiO_2$), 10 kV



Solution overlaid on the diffraction pattern giving the crystal orientation as {370}<7-34>



Simulated diffraction pattern showing all Kikuchi bands with intensity greater than 10% of the most intense band.



Simulation of crystal orientation

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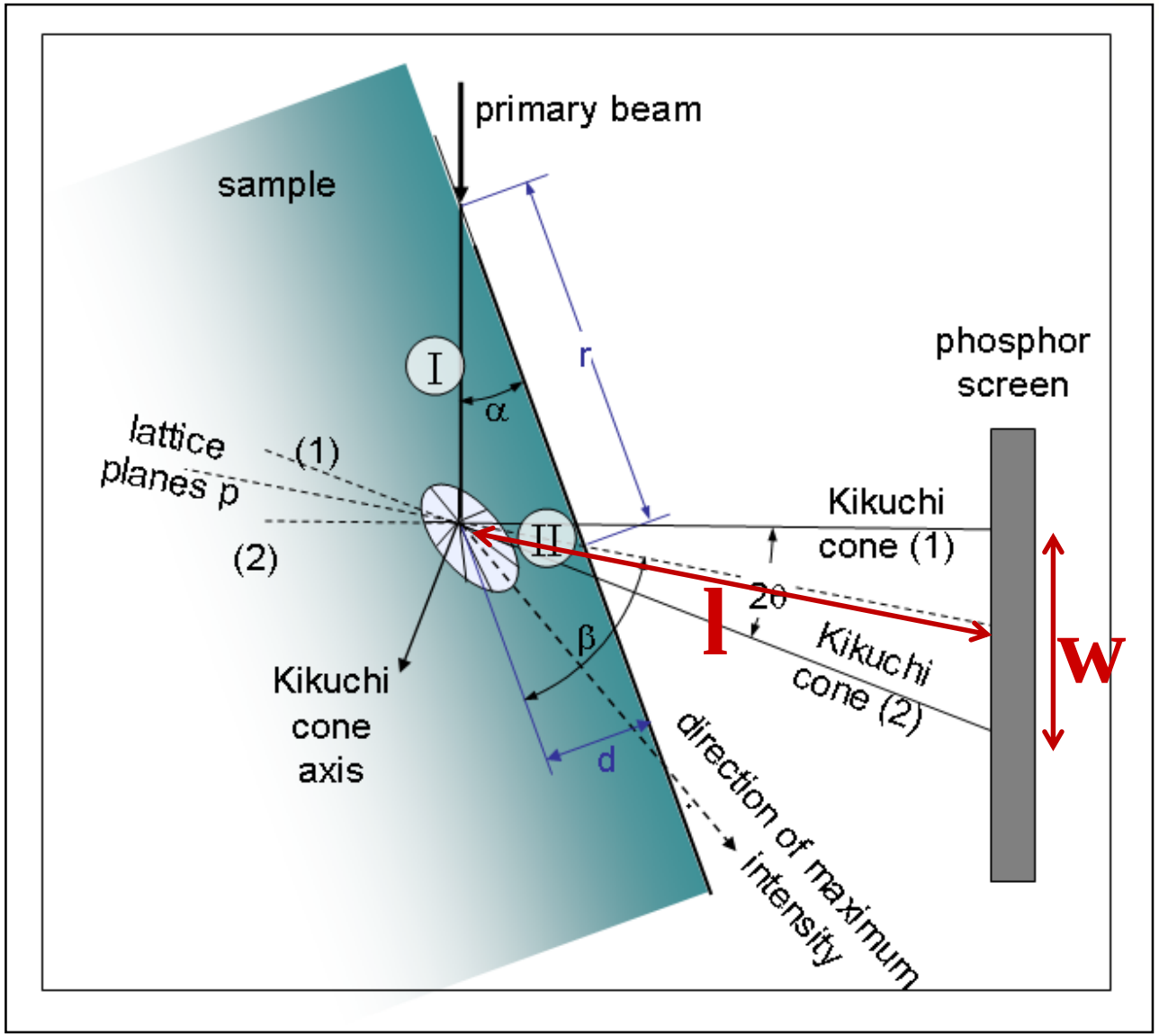

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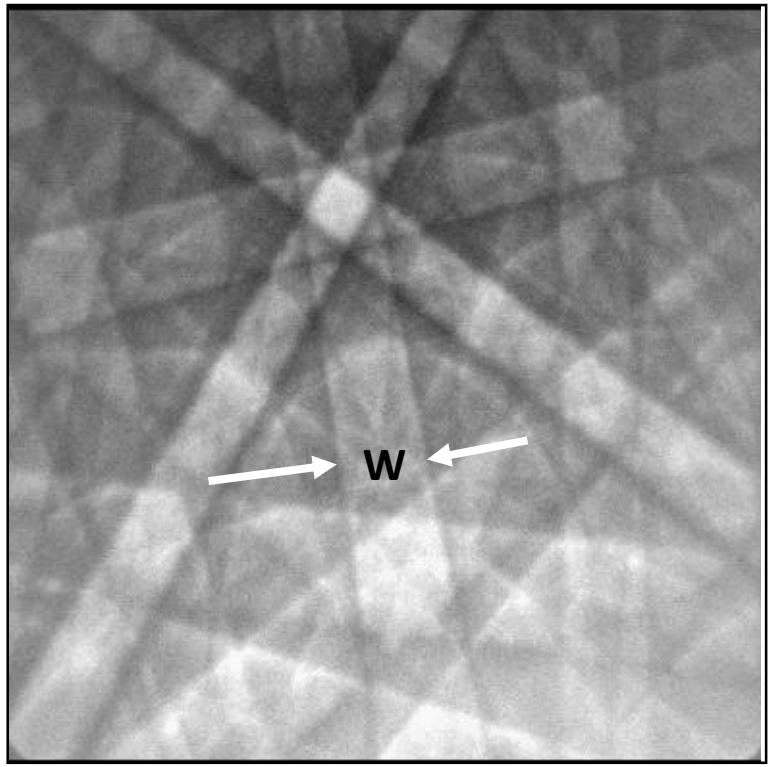
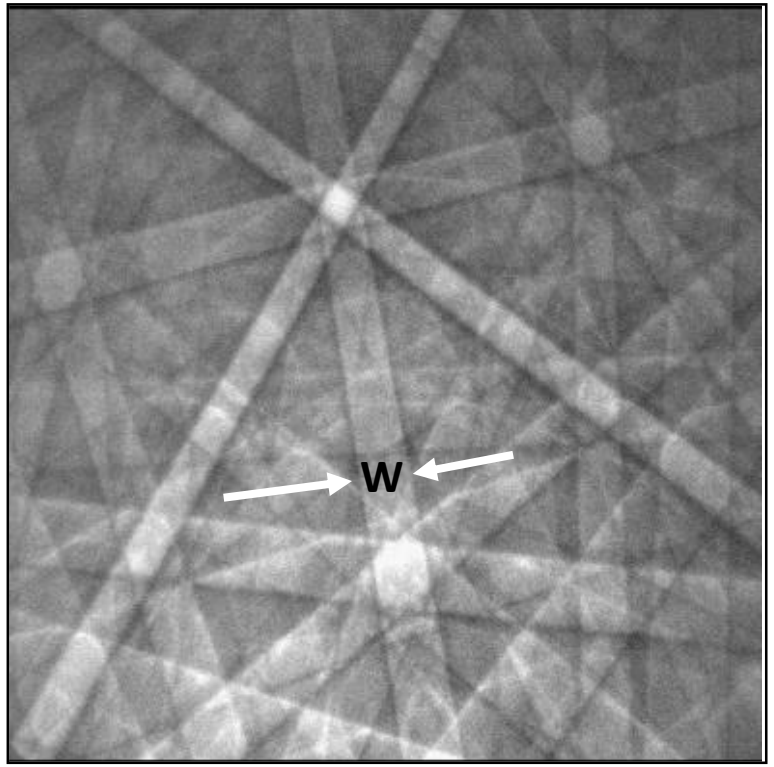
Bragg's Law – Voltage Effect

$$\lambda = 2d_{hkl} \sin\theta$$

$$n\lambda = 2d_{hkl} \sin\theta = 2d_{hkl}\,\theta$$

$$w = 2l \sin\theta = 2l\theta$$





$$w = \frac{l\lambda}{d}$$

EBSPs from silicon at 10 keV and 30 keV

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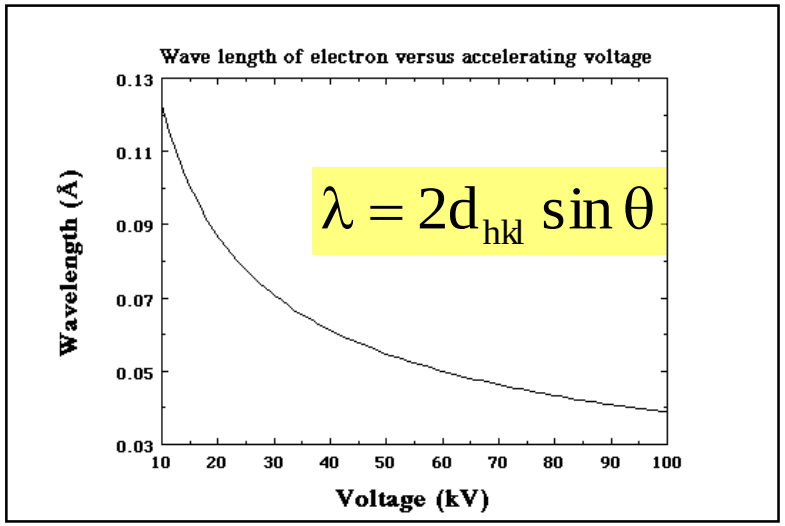
$$\lambda = \frac{h}{\sqrt{2m_o eV\left[\frac{1 + eV}{2m_o c^2}\right]}}$$

$$\lambda = \frac{12.2630}{\sqrt{V + 0.97845 \times 10^{-6} V^2}}$$



V (kV)	λ, nm
20	0.00859
30	0.00698
40	0.00602
50	0.00536
60	0.00487
70	0.00448
80	0.00418
90	0.00392
100	0.00370
200	0.00251
300	0.00197
400	0.00164
500	0.00142
600	0.00126
700	0.00113
800	0.00103
900	0.00094
1000	0.00087
2000	0.00050
4000	0.00028

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Summary (1)

1. When an electron beam hits a tilted crystalline sample, electron backscatter diffraction patterns are formed on a suitably placed phosphor screen.
2. Diffraction pattern consists of a set of Kikuchi bands which are characteristic for the sample crystal structure and orientation.
3. The center line of each Kikuchi band corresponds to the intersection with the phosphor screen of the diffracting plane responsible for the band.
4. The position of the Kikuchi bands can be found automatically with the Hough transform and used to calculate the orientation of crystal that formed the pattern.

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3. EBSD – solving the pattern

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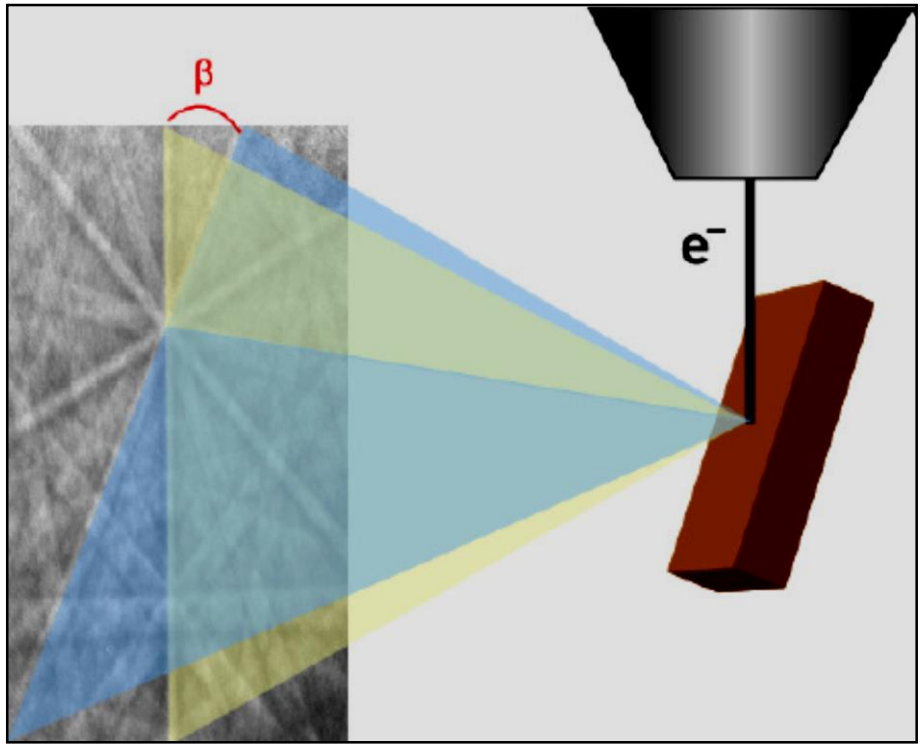
If the *hkl* of two bands in the pattern can be identified then the corresponding orientation can be calculated.

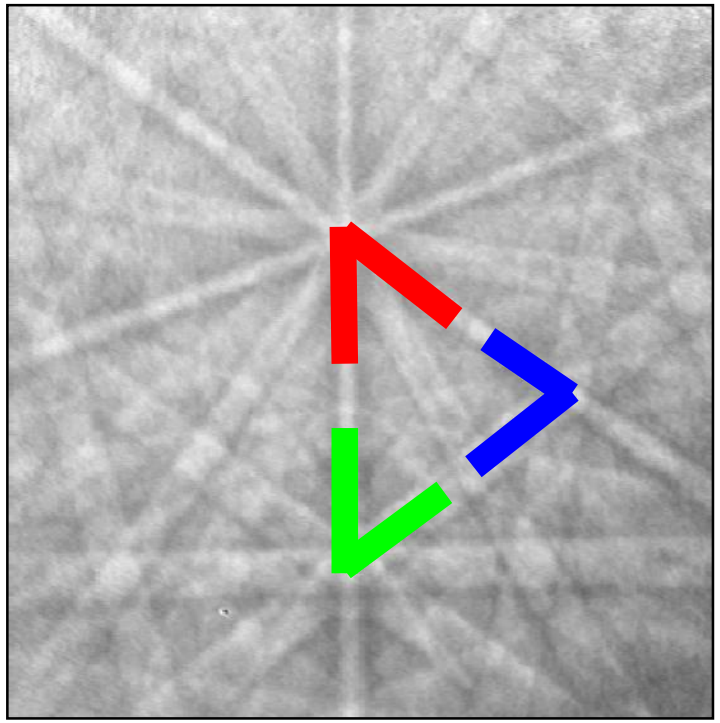
By comparing the angle between two bands with an interplanar angle in Look-up table, the *hkl* pair associated with the band pair can be identified.

If 3 bands are used then the *hkls* associated with all 3 bands can be determined.



Angle	$(hkl)_1$	$(hkl)_2$	Angle	$(hkl)_1$	$(hkl)_2$
25.2	200	311	64.8	220	$3\bar{1}1$
29.5	111	311	70.5	111	$11\bar{1}$
31.5	220	311	72.5	200	131
35.1	311	$31\bar{1}$	80.0	111	$3\bar{1}\bar{1}$
35.3	111	220	84.8	311	$1\bar{3}1$
45.0	200	220	90.0	111	$2\bar{2}0$
50.5	311	$3\bar{1}\bar{1}$	90.0	200	020
54.7	111	200	90.0	200	022
58.5	111	$31\bar{1}$	90.0	220	$11\bar{3}$
60.0	220	202	90.0	220	$2\bar{2}0$
63.0	311	$13\bar{1}$			

A set of orientations is obtained from triplet of bands by comparing the interplanar angles against a Look-up table



Angle	(hkl)1	(hkl)2
25.2	200	311
29.5	111	311
31.5	220	311
35.1	311	311
35.3	111	220
45.0	200	220
50.5	311	311
54.7	111	200
58.5	111	311
60.0	220	202
63.0	311	131
64.8	220	311
70.5	111	111
72.5	200	131
80.0	111	311
84.8	311	131
90.0	111	220
90.0	200	020
90.0	200	022
90.0	220	113
90.0	220	220

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Building an interplanar Look-up table

1) Identify the *hkl* of the high contrast bands (bands likely to be detected by the Hough transform).

200
111
220
311

2) Determine all of the symmetrically equivalent *hkl*'s.

$200, 020, 002$
$111, 11\bar{1}, 1\bar{1}1, \bar{1}11$
$220, 2\bar{2}0, 202, 20\bar{2}, 022, 02\bar{2}$
$311, \bar{3}11, 3\bar{1}1, 31\bar{1}, 131, \bar{1}31, 1\bar{3}1, 13\bar{1}, 113, \bar{1}13, 1\bar{1}3, 11\bar{3}$

3) Form all possible pairs.

$200, 020 \quad 200, 002 \quad 200, 111 \quad 200, 11\bar{1} \quad \cdots$
$020, 002 \quad 020, 111 \quad 020, 11\bar{1} \quad 020, 1\bar{1}1 \quad \cdots$
$002, 111 \quad 002, 11\bar{1} \quad 002, 1\bar{1}1 \quad 002, \bar{1}11 \quad \cdots$
$\vdots$

4) Calculate the angles between the plane pairs.

$200\angle 020 = 90° \quad 200\angle 002 = 90° \quad 200\angle 111 = 54.7° \quad 200\angle 11\bar{1} = 54.7° \quad \cdots$
$020\angle 002 = 90° \quad 020\angle 111 = 54.7° \quad 020\angle 11\bar{1} = 54.7° \quad 020\angle 1\bar{1}1 = 54.7° \quad \cdots$
$002\angle 111 = 90° \quad 002\angle 11\bar{1} = 90° \quad 002\angle 1\bar{1}1 = 90° \quad 002\angle \bar{1}11 = 90° \quad \cdots$
$\vdots$

5) Throw out duplicates and sort.

$200\angle 311 = 25.2° \quad 111\angle 220 = 35.3° \quad 111\angle \bar{3}11 = 58.5° \quad 111\angle 11\bar{1} = 70.5°$
$111\angle 311 = 29.5° \quad 200\angle 220 = 45.0° \quad 220\angle 202 = 60° \quad 200\angle 131 = 72.5°$
$220\angle 311 = 31.5° \quad 311\angle \bar{3}11 = 50.5° \quad 311\angle 13\bar{1} = 63.0° \quad 111\angle \bar{3}11 = 80°$
$311\angle \bar{3}11 = 35.1° \quad 200\angle 111 = 54.7° \quad 220\angle 3\bar{1}1 = 64.8° \quad \vdots$

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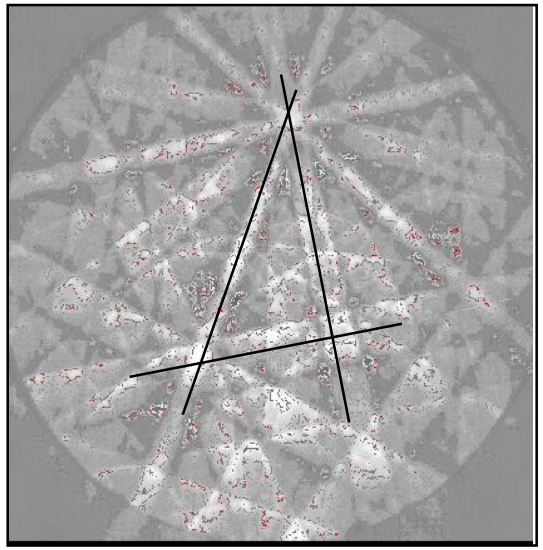
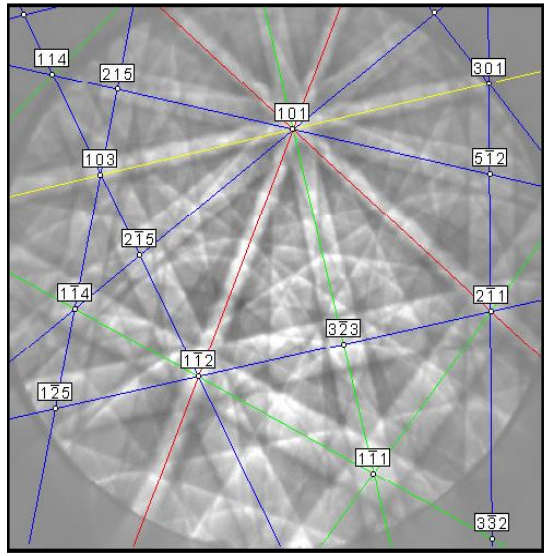
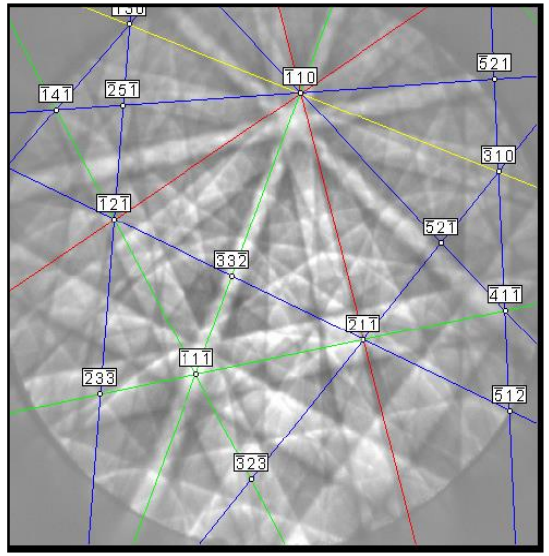
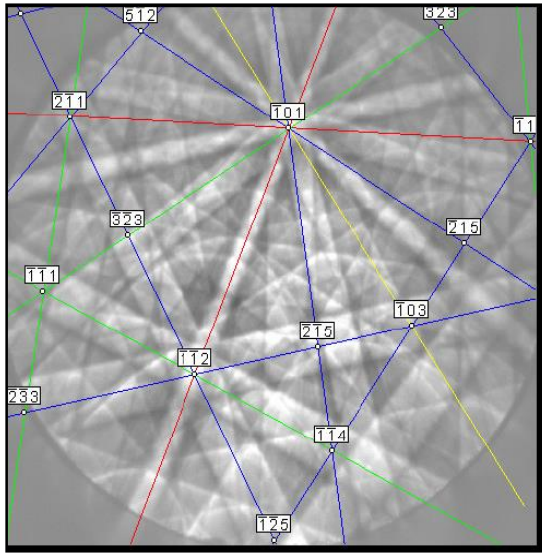


For a set of three bands, compare the interplanar angles against the Look-up table and determine all possible indexing solution







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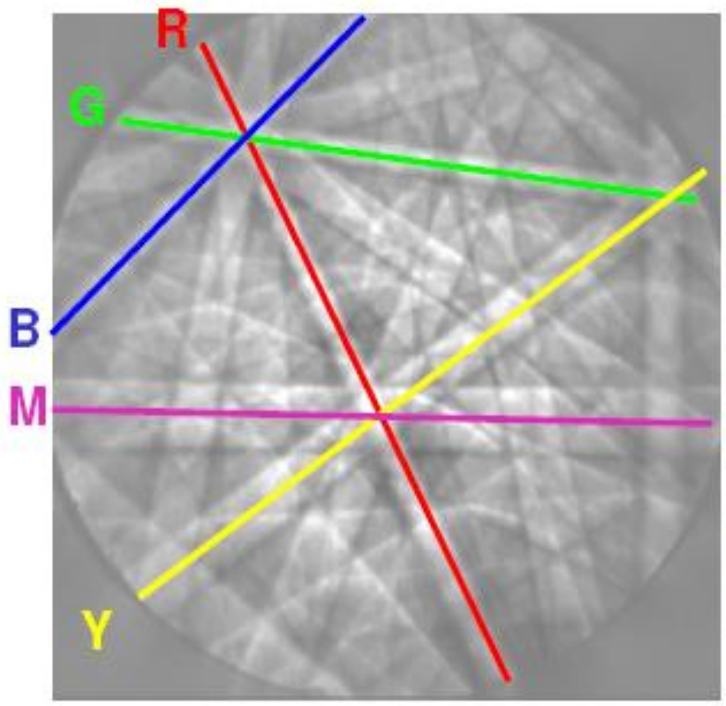
$$\#triplets = \frac{n!}{(n-3)!\cdot 3!}$$

For a given number of bands, *n*, used for pattern indexing, the number of band triplets is determined by this formula.

Typically 7 to 9 detected bands are used for automatic indexing.

n	$\#\ triplets$
3	1
4	4
5	10
6	20
7	35
8	56
9	84



Triplet	Solution 1 (V_1)	Solution 2 (V_2)	Solution 3 (V_3)
R, G, Y	X		
R, G, B	X		
R, G, M	X		
R, Y, B	X		
R, Y, M	X	X	
R, B, M	X		
G, Y, B	X		
G, Y, M	X		
G, B, M	X		
Y, B, M	X		X
Total	10	1	1

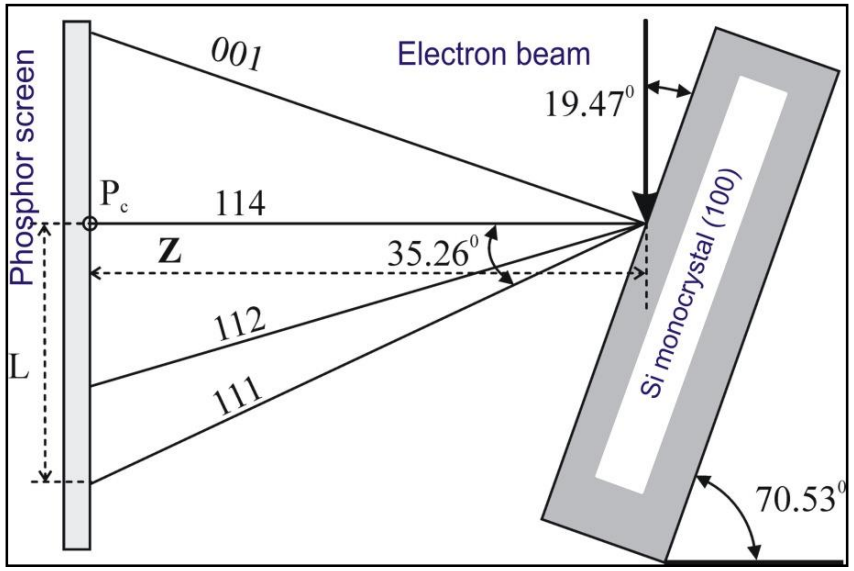
For this set of 5 detected bands, 10 triplet combinations are possible.

For each of these 10 triples solution V_1 matched.

Solutions V_2 and V_3 each matched one triplet only.

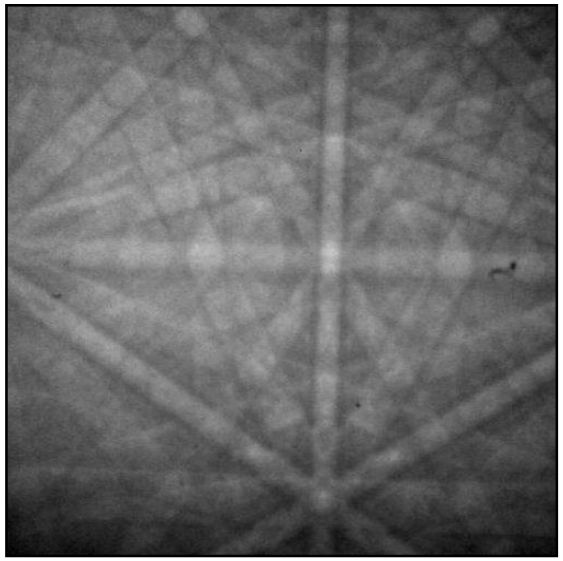
Confidence Index (CI - TSL) – information whether the matching is good or bad

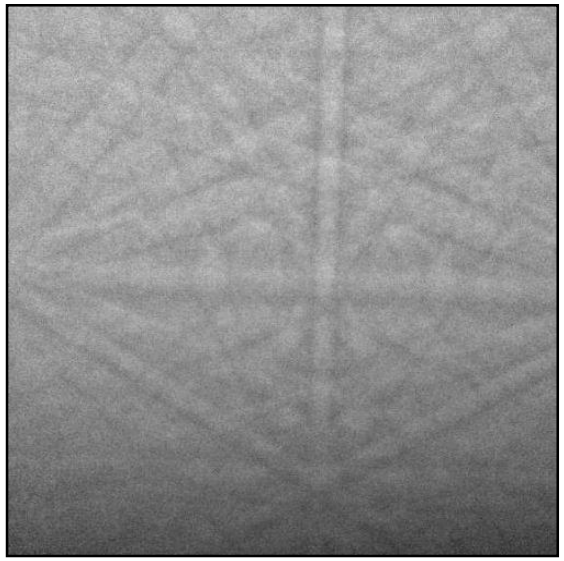
Pattern Centre – why do we tilt up to 70°?



The pattern centre (PC) is the point on the screen closest to the generation point of the diffraction pattern on the sample

Tilt 70°





Tilt 50°

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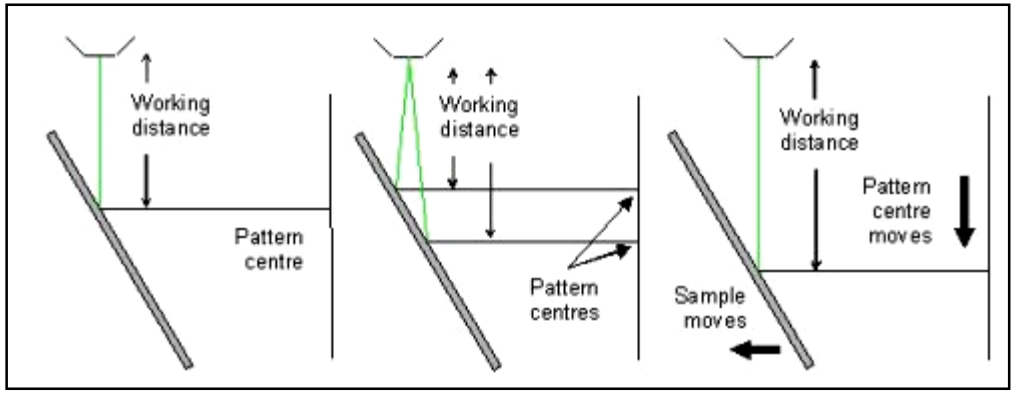
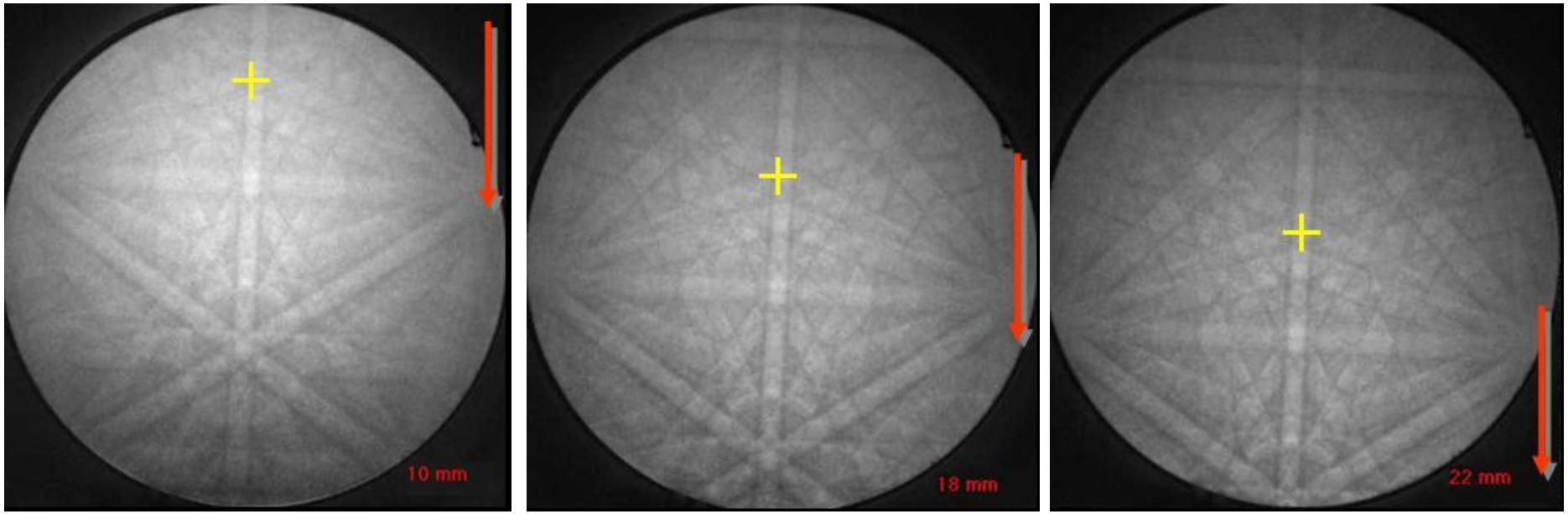


As the working distance changes the pattern centre changes as well



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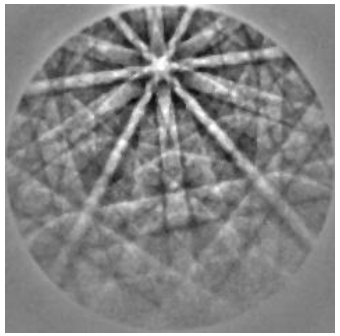
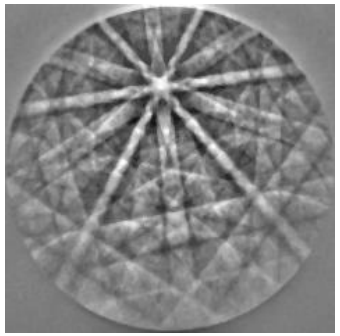
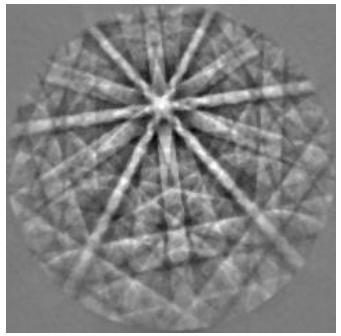


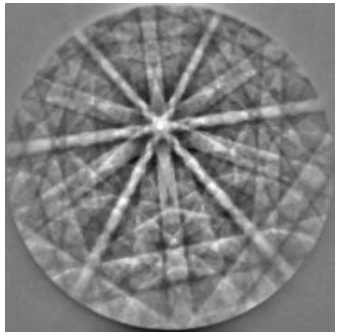
WD 7 mm



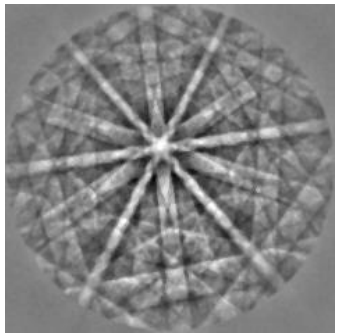
WD 9 mm



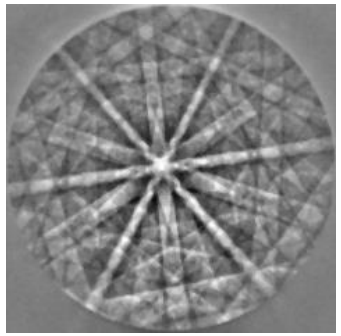
WD 11 mm



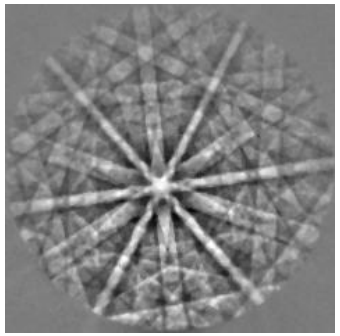
WD 13 mm



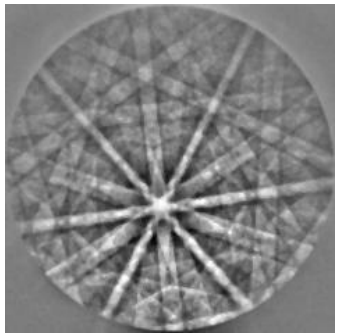
WD 15 mm



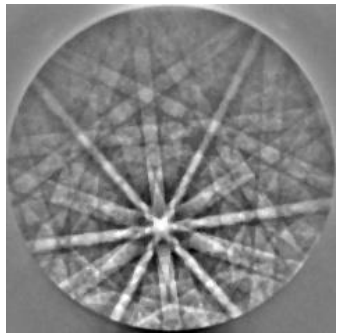
WD 17 mm



WD 19 mm



WD 21 mm



WD 23 mm

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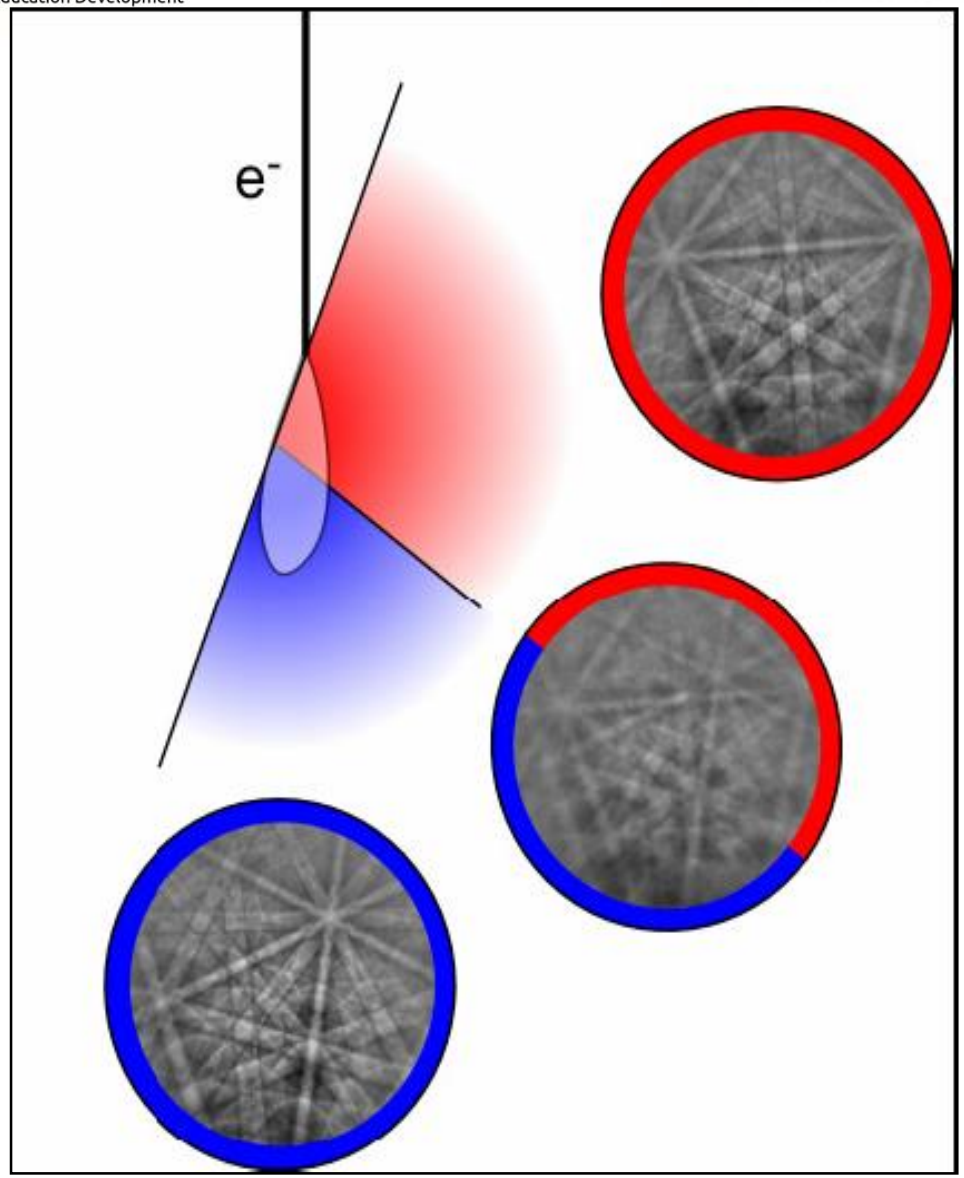

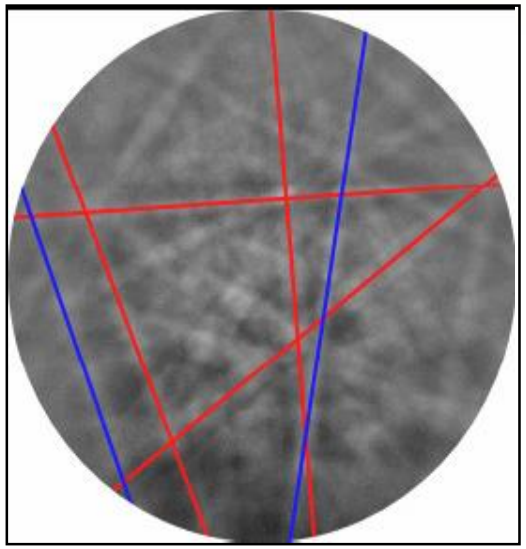

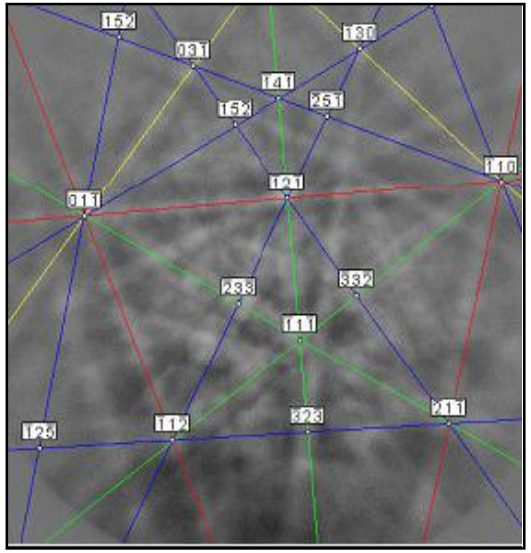

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4. Information available from EBSD

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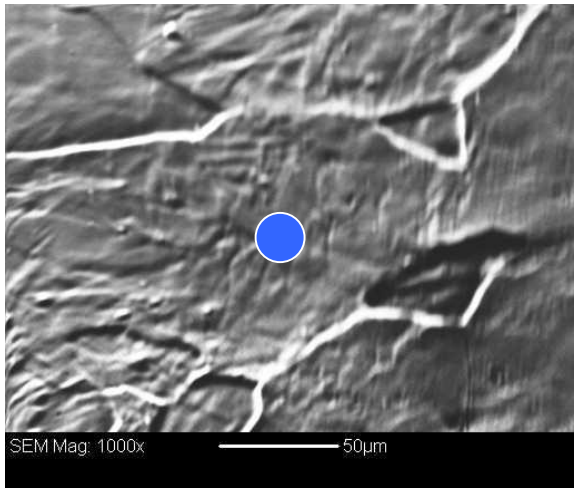
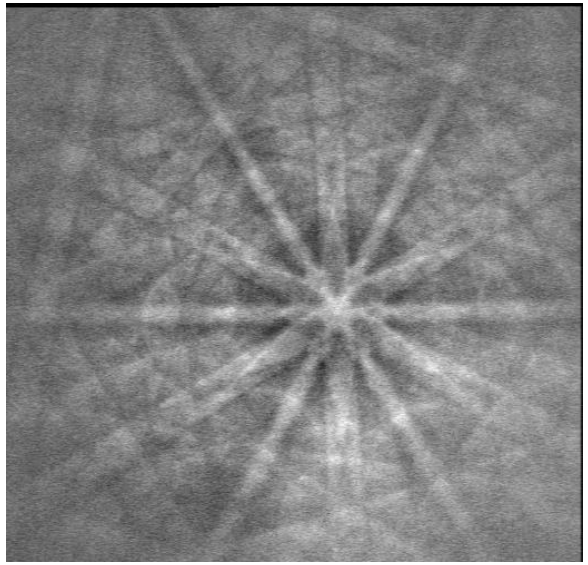
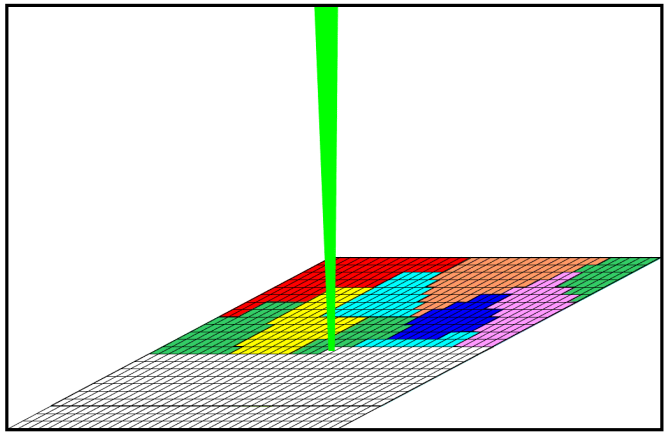
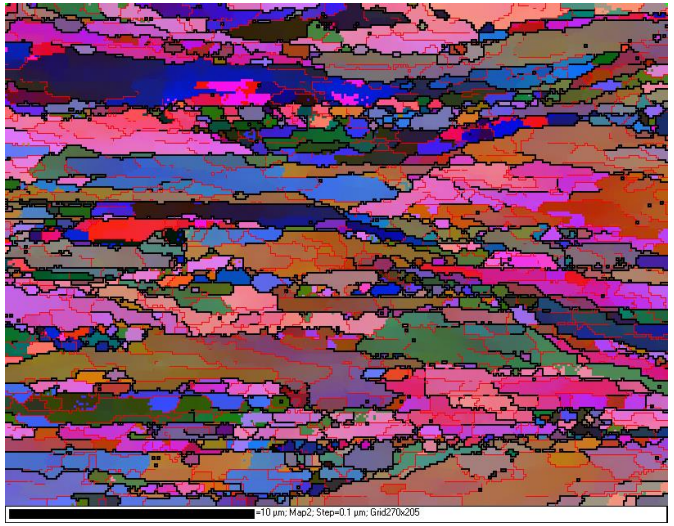
Point analysis





Scan analysis





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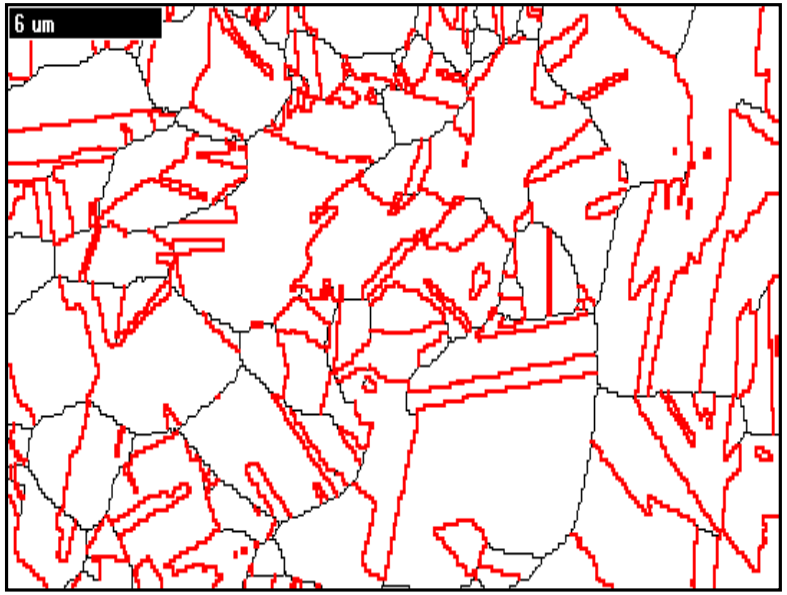
Grain boundaries in brass
All Euler angles map



Coincidence Site Lattice (CSL) boundaries in brass Σ3 (twins) – 67% red lines

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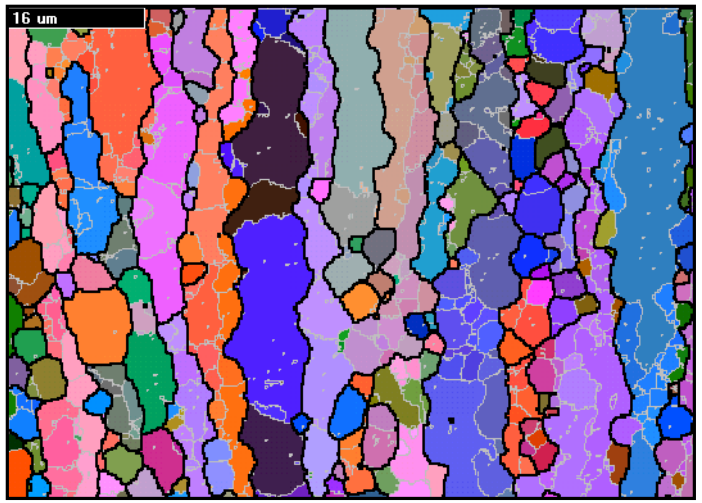
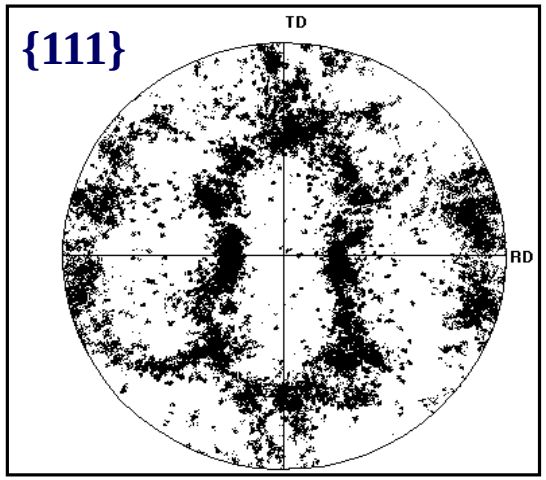
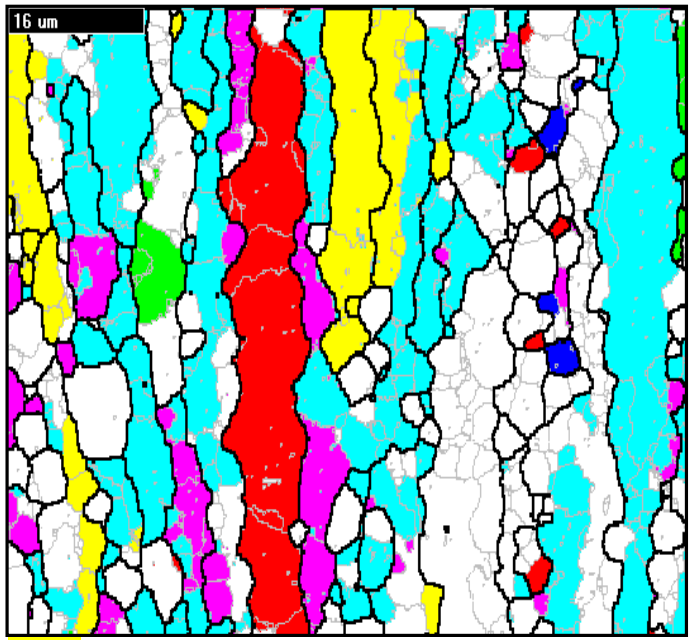
The „preferred orientation”, i.e. the non-random orientation of single crystal lattices within a materials usually obtained by its processing







Brass {011}<112>
Copper {112}<111>
Cube {001}<100>
Goss {011}<100>
P {011}<122>
S {123}<634>

Hot rolled AA5182 alloy

Texture Analysis

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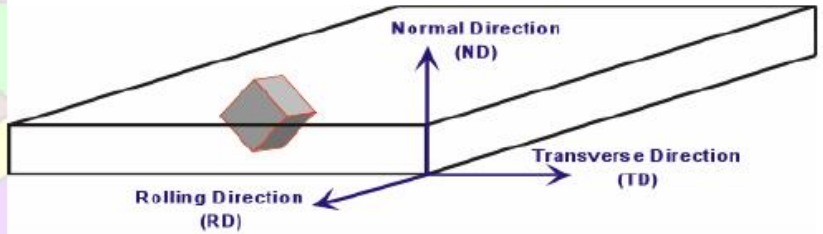
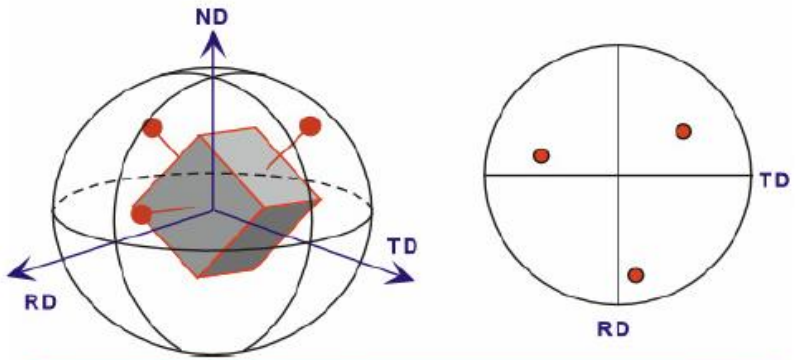
Pole Figures

Consider a cubic crystal in a rolled sheet sample with "laboratory" or "sample" axes as shown below.



The Pole Figure plots the orientation of a given plane normal (pole) with respect to the sample reference frame. The example below is a (001) pole figure. Note the three points shown in the pole figure are for three symmetrically equivalent planes in the crystal.



TSL

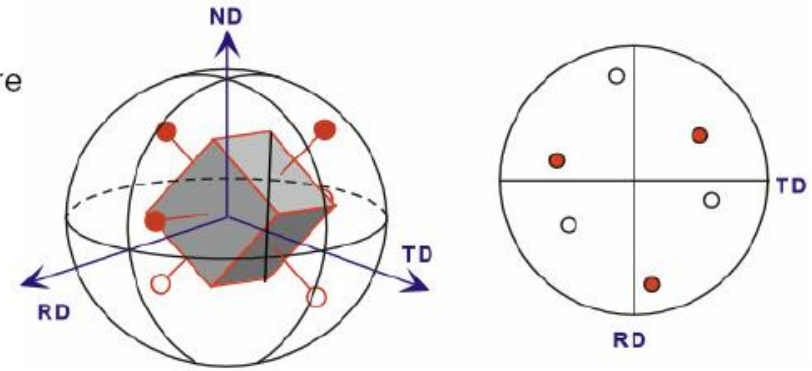
EDAX TSL
advanced microanalysis solutions

Pole Figures: Negative Hemisphere

Positive hemisphere

Negative Hemisphere

Open circles can be used to distinguish points in the negative hemisphere from points in the positive hemisphere in the same pole figure.

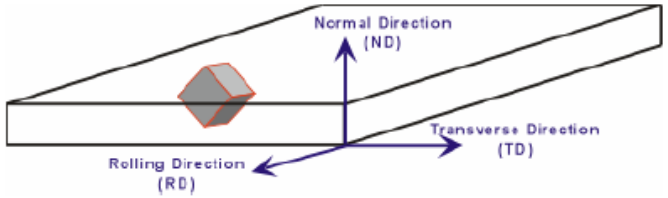


TSL

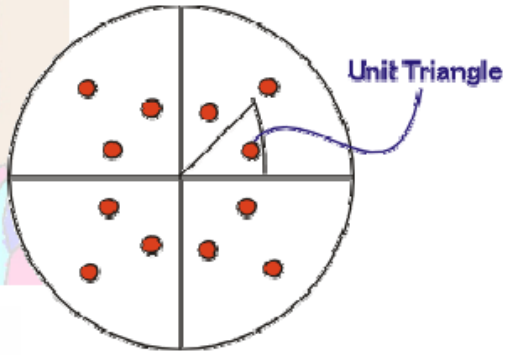
EDAX TSL

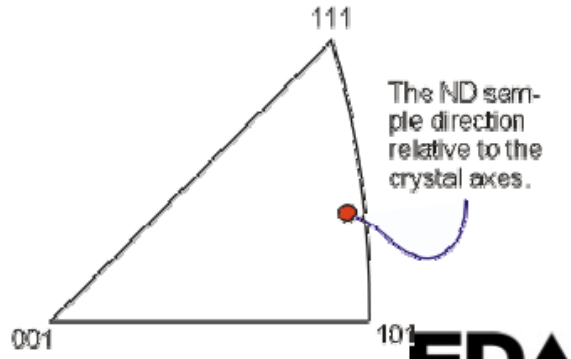
Inverse Pole Figures

Consider a cubic crystal in a rolled sheet sample with "laboratory" or "sample" axes as shown below.



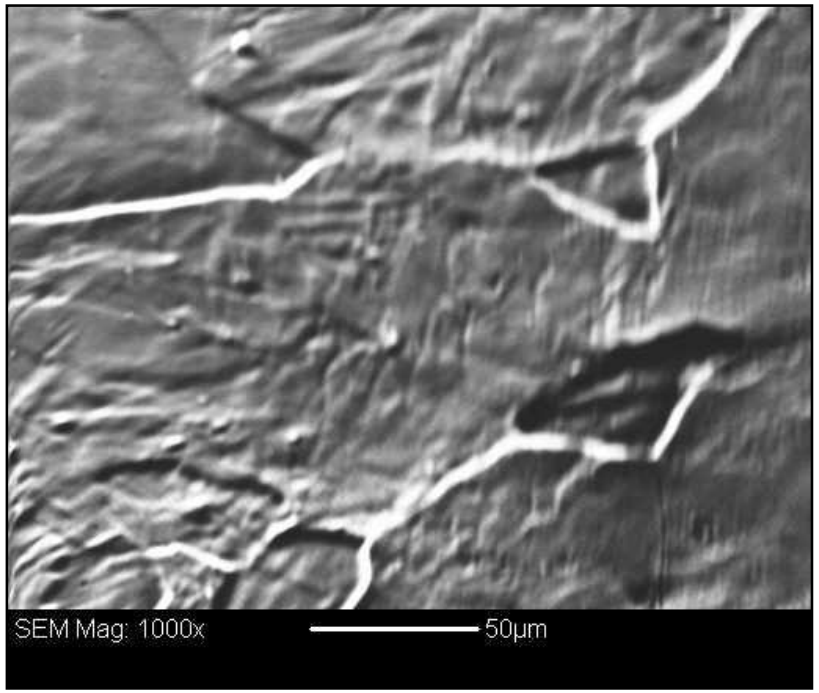
There are two ways of looking at inverse pole figures: 1) Which crystal axis is aligned with a specified sample axis. 2) The orientation of the specified sample axis with respect to the crystal axes. The example below is a normal direction inverse pole figure. In the full inverse pole figure all symmetrically equivalent points are shown.

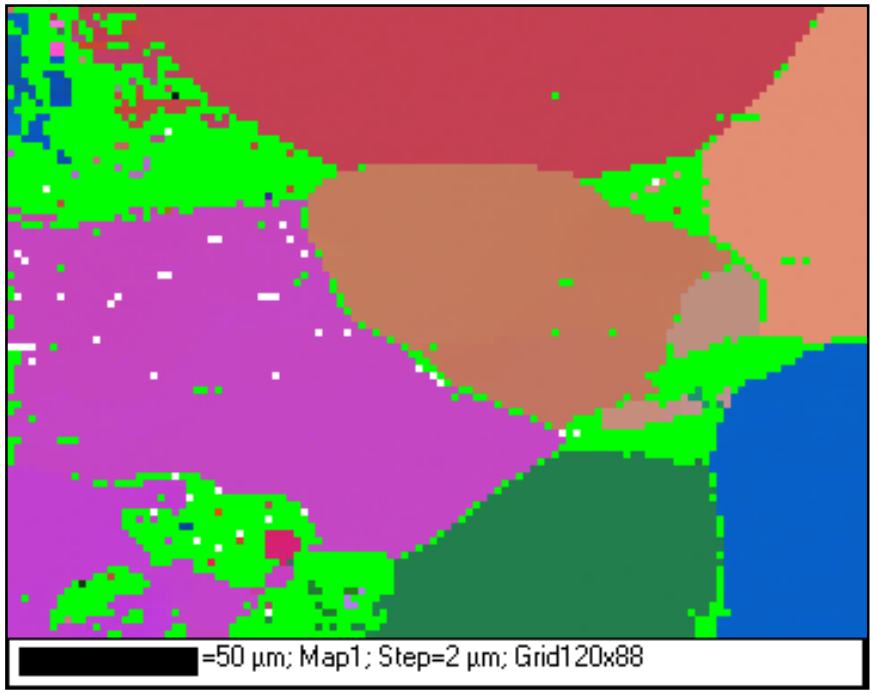




TSL EDAX TSI

Fe0.05wt%C 725°C 554h





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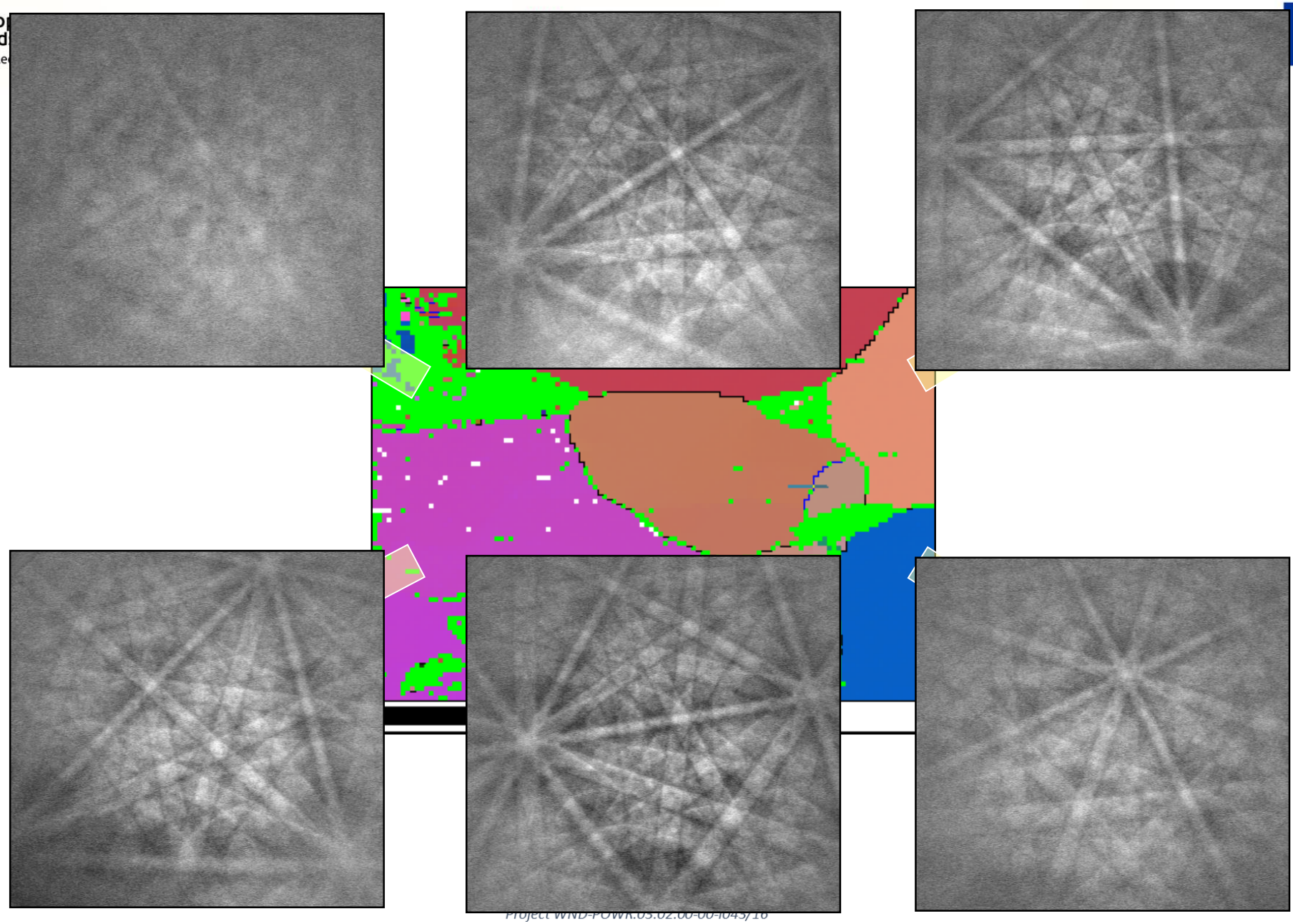
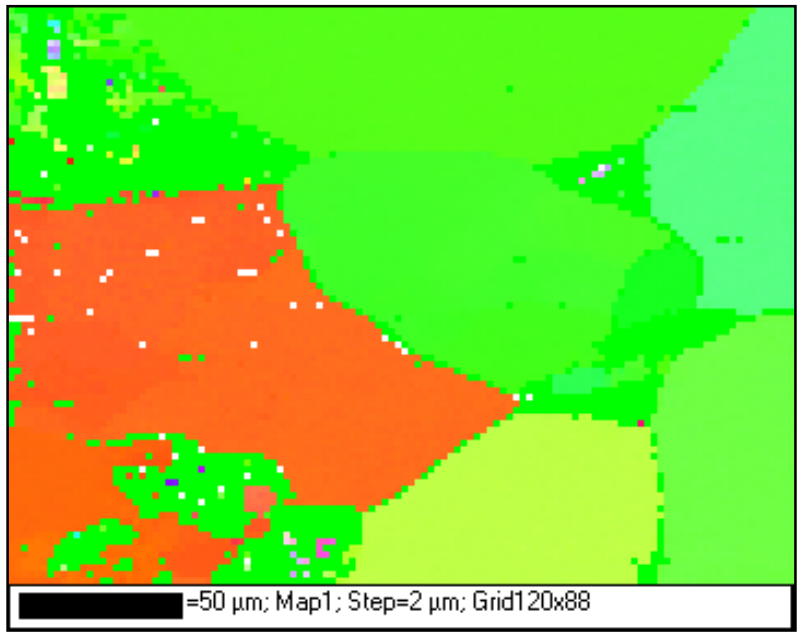
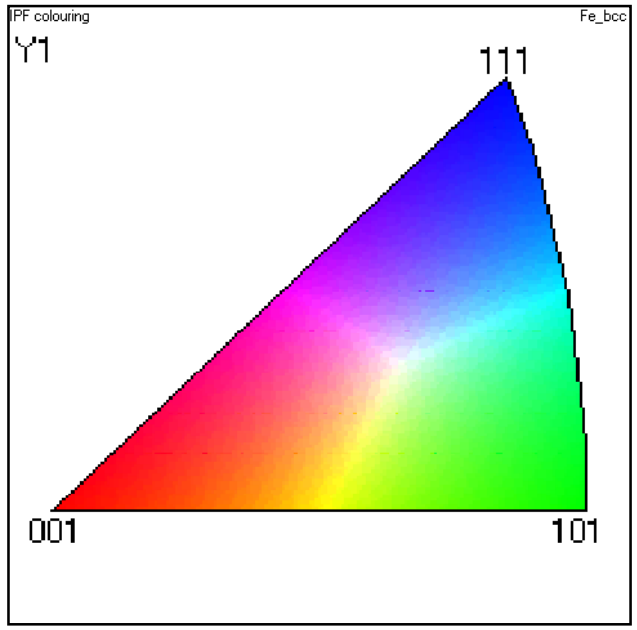


Fe0.05wt%C 725°C 554h





OIM Inverse Pole Figure Map

Color coding indicates crystal direction parallel to a reference direction (here // to transverse direction)

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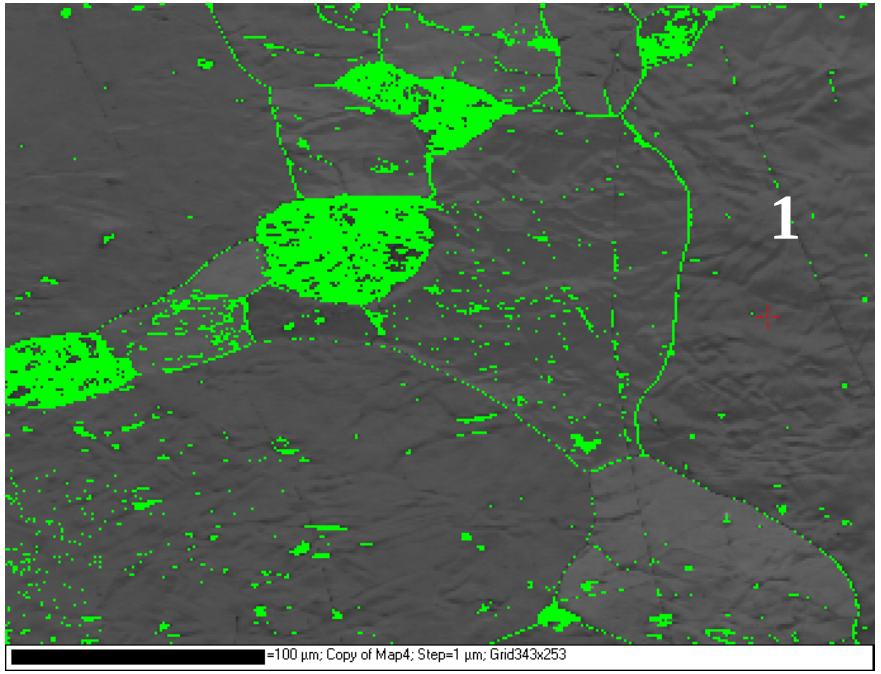
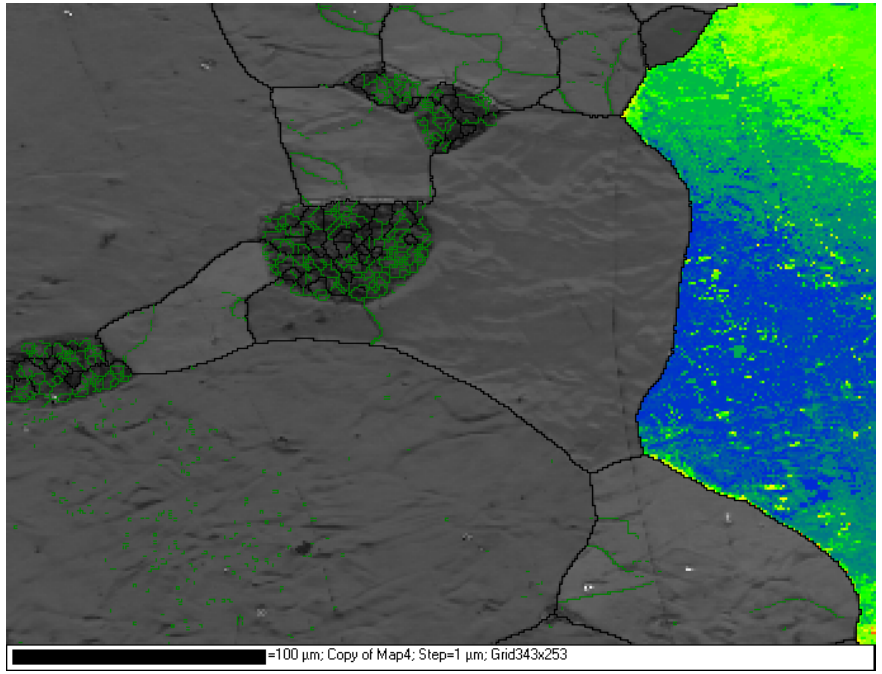
Fe0.05wt%C 725°C 554h



BC map (*Band Contrast)*



TC map (*Texture Component Map*)
Ideal orientation grain 1:
ϕ_1=130,6° , Φ=40,1°, ϕ_2=67,0°,
deviation from ideal orientation 1°

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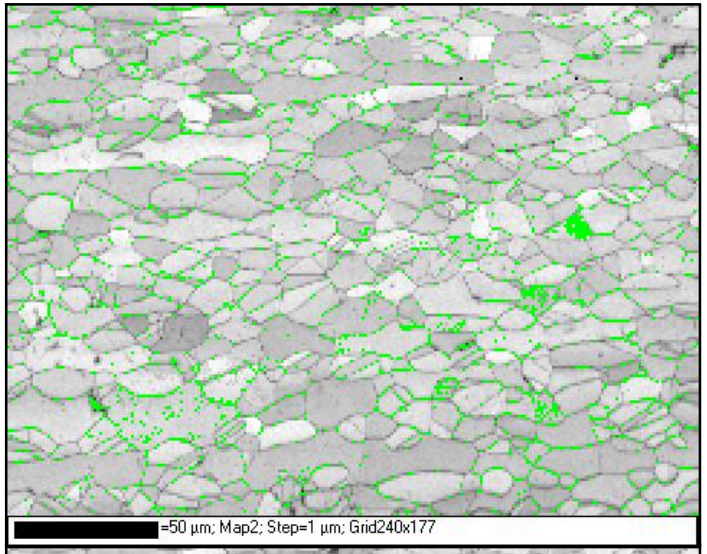
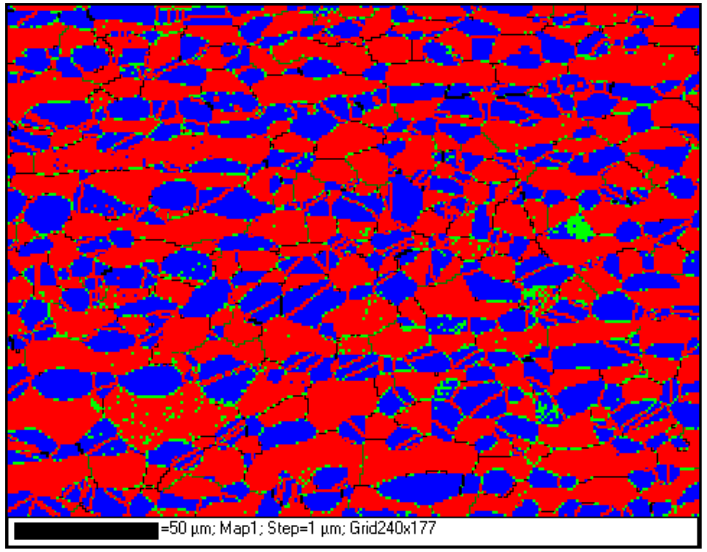
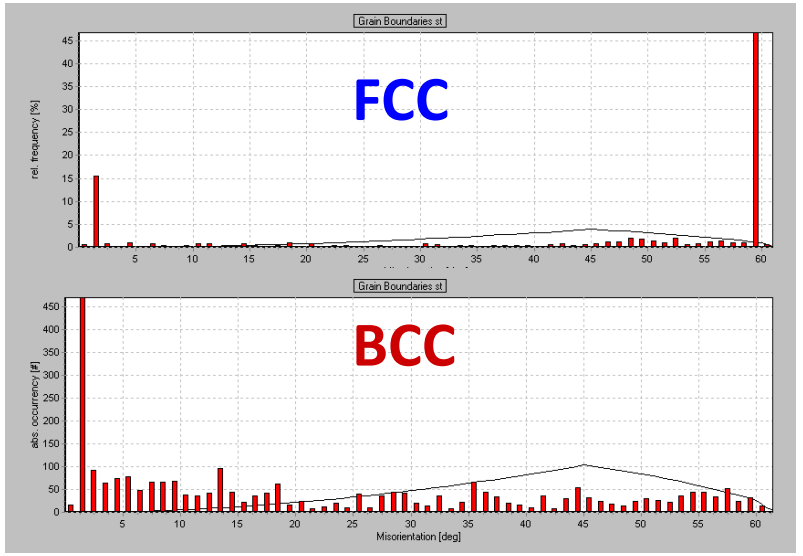
DUPLEX Steel

BC map (*Band Contrast +Noise)*



BC map after noise reduction





Phase map + boundaries distribution

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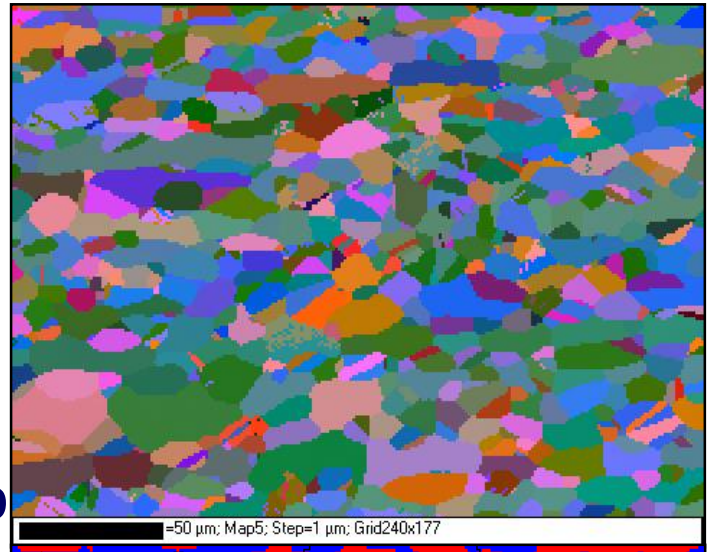
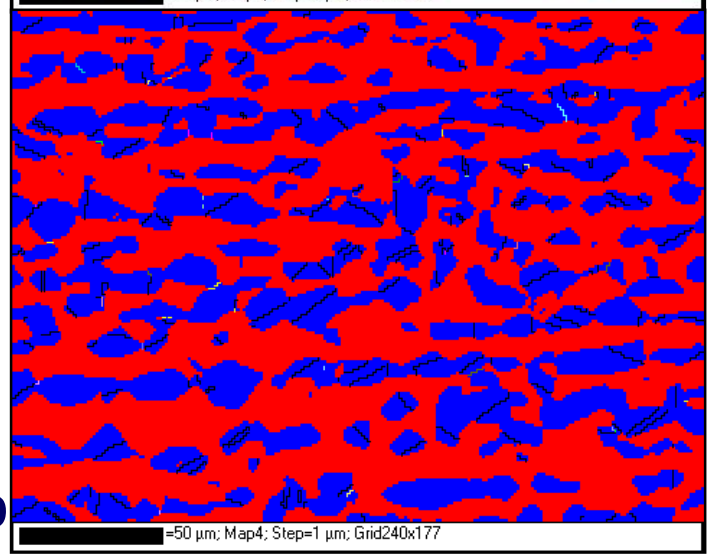
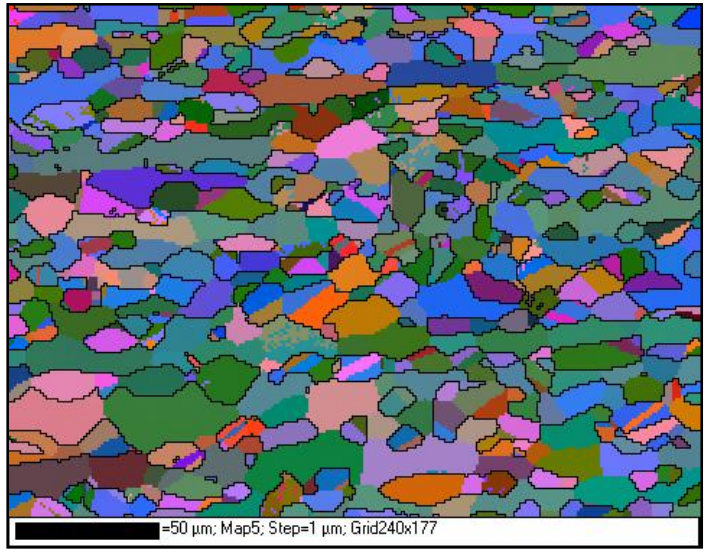
Orientation map

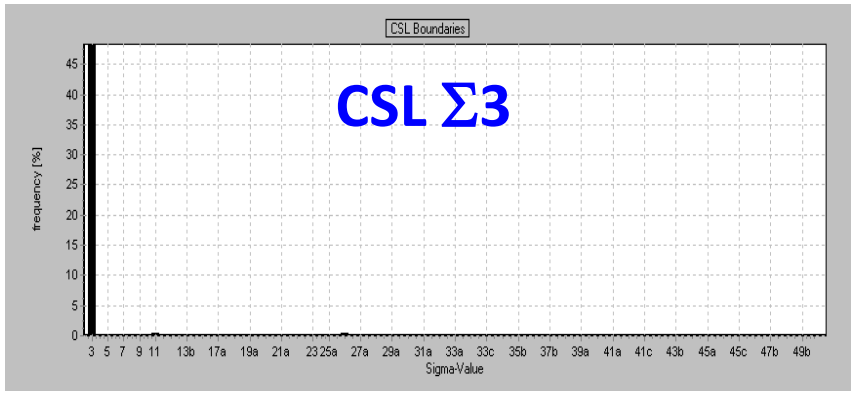


Phase map





Orientation map + grain boundaries



CSL boundary – the description of a grain boundary by a specific coincidence of the crystal lattices across the boundary. The most common CSL boundaries (e.g. Σ 3, Σ 9, and Σ 27) are twin boundaries

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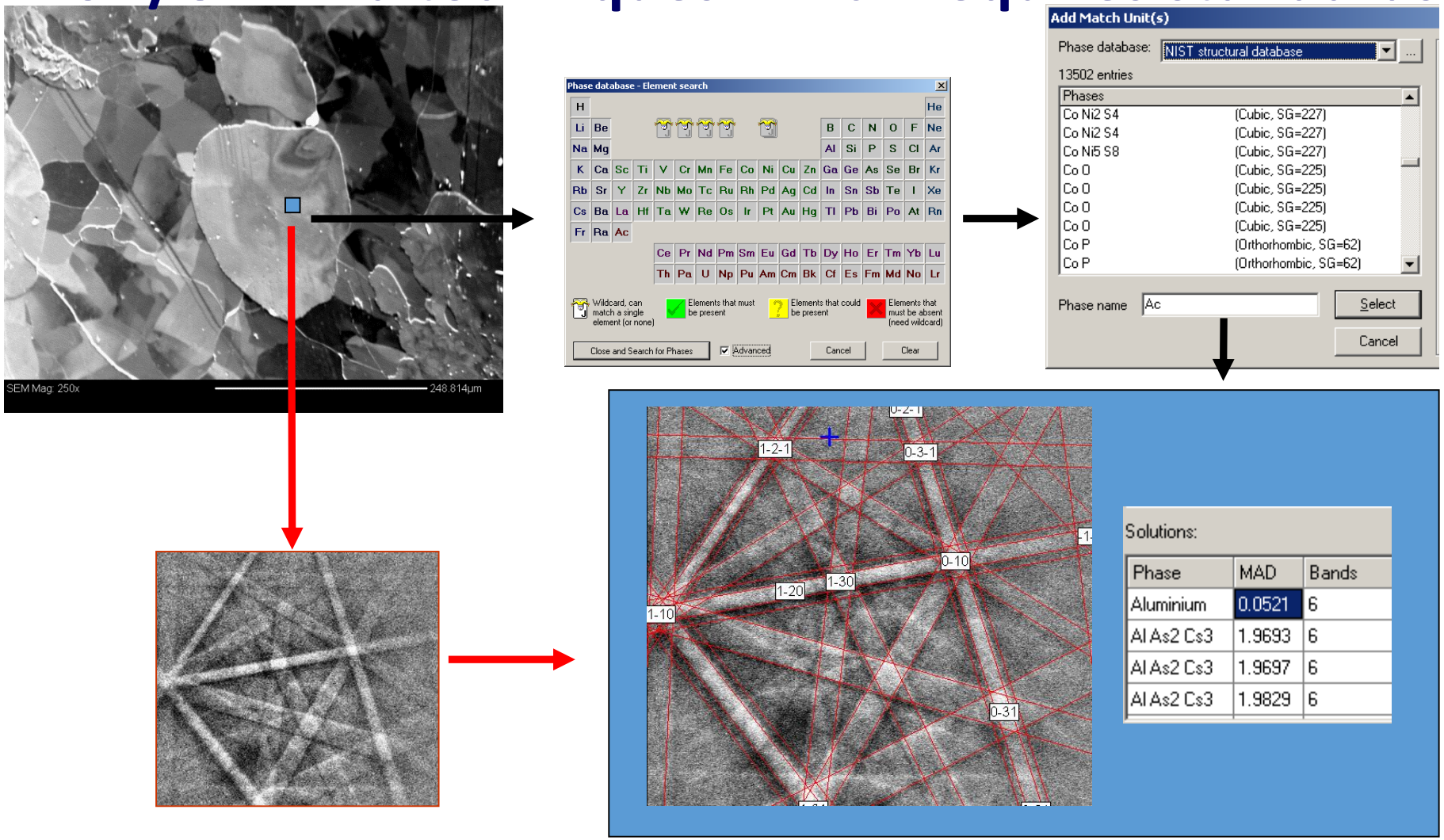
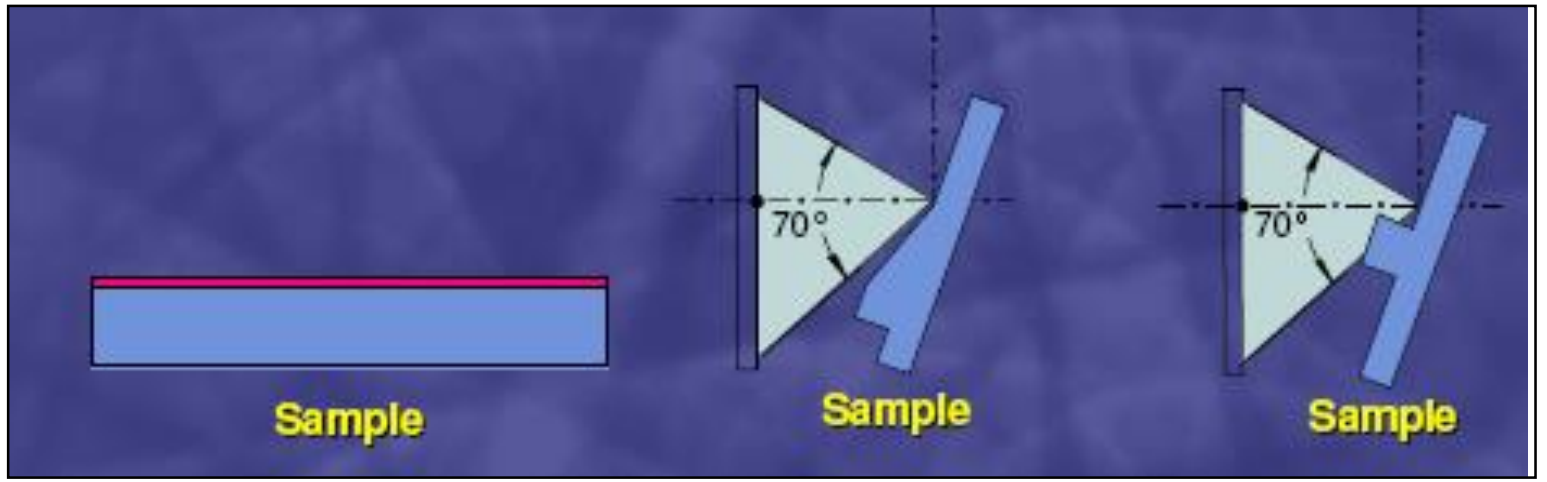
EBSD/OIM – a techniques which requires standards



Sample preparation



Specimen requirements for EBSD analysis:

Information depth is ~50 nm therefore:

- **Crystal structure should be continuous up the specimen surface**
- **No deformation layer, no oxidation layer, no coating**
- **Smooth surface layer only required to avoid shadowing**

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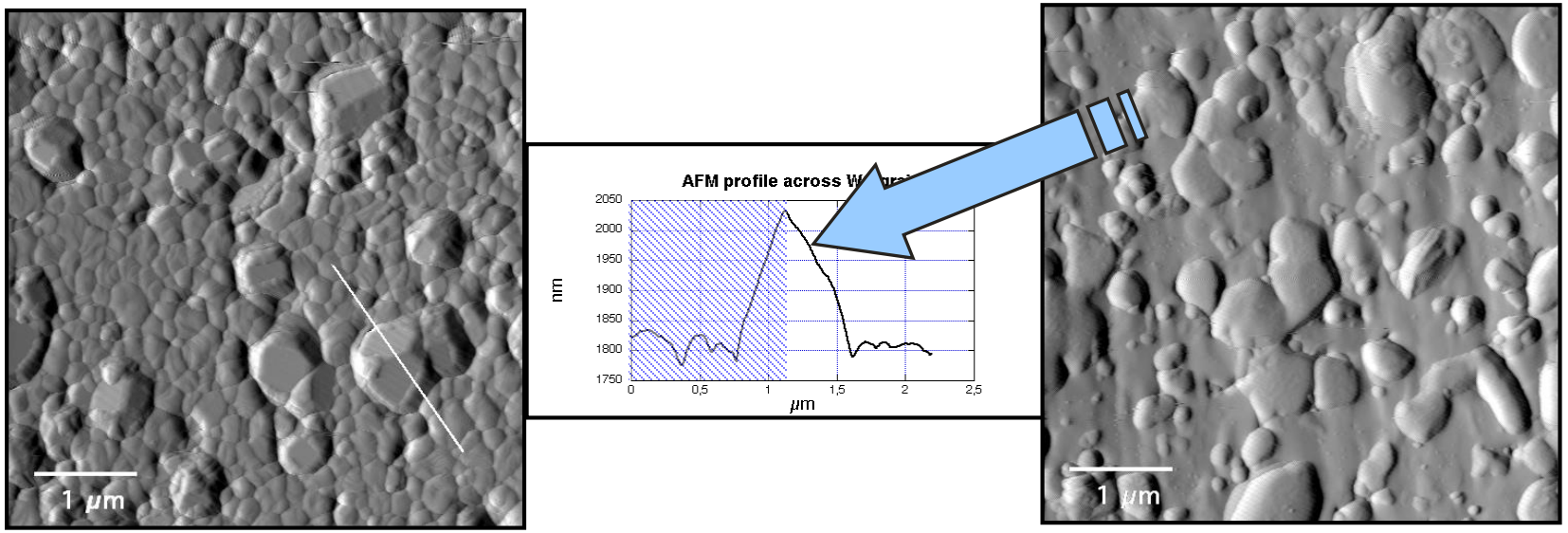
Y-TZP (0,2 – 0,3 µm) /WC composite



Thermal etching

Mechanical polishing by use of colloidal silica

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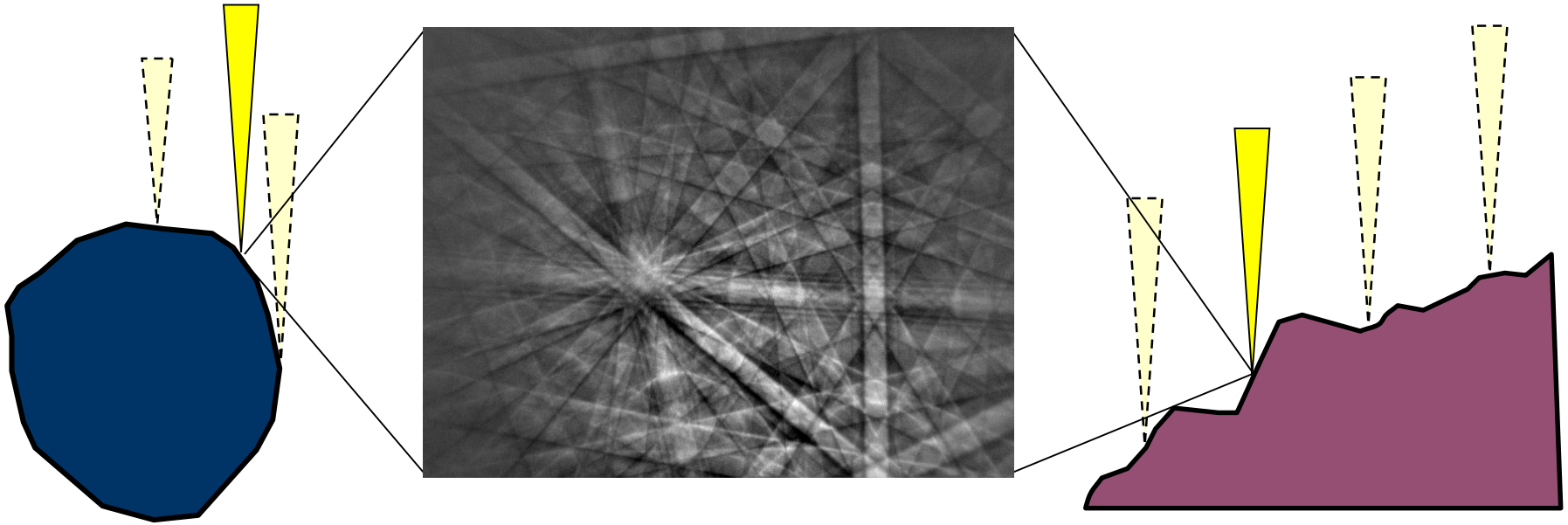

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5. A few examples

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Phase map from emery rock sample

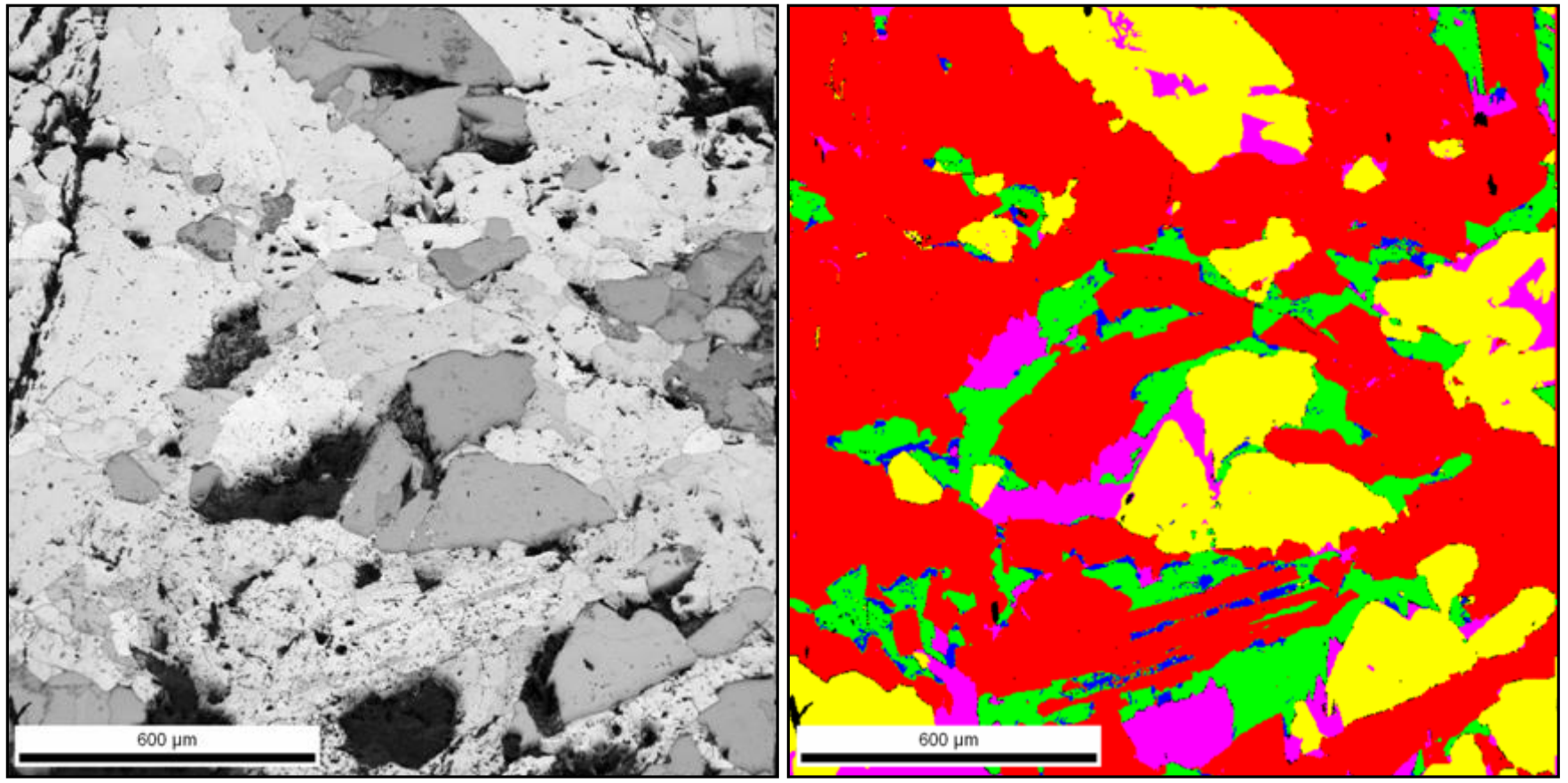

Image quality based on Band Contrast (left) and Phase Map (right)

Courtesy of EDAX

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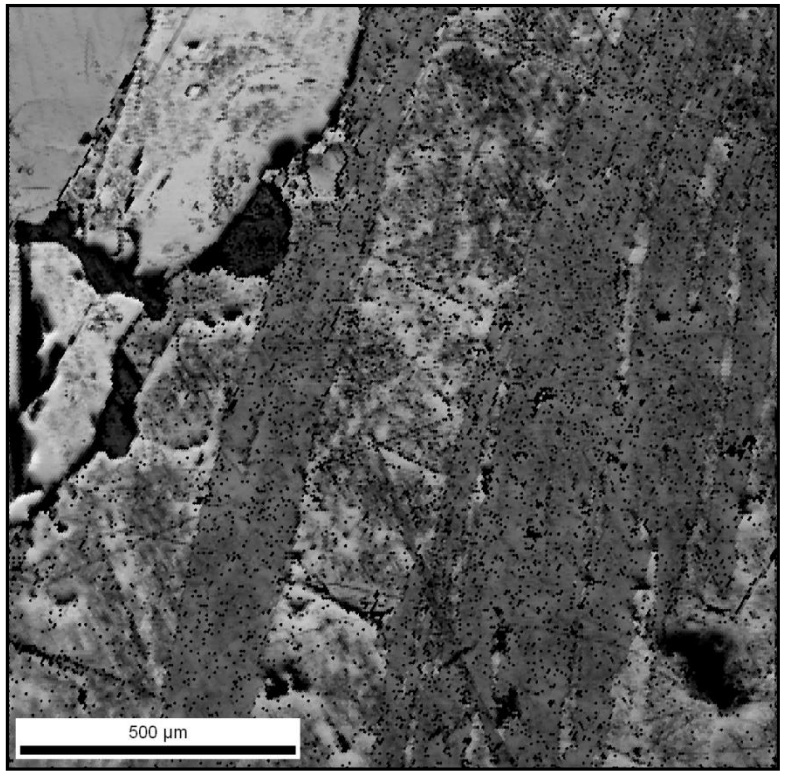

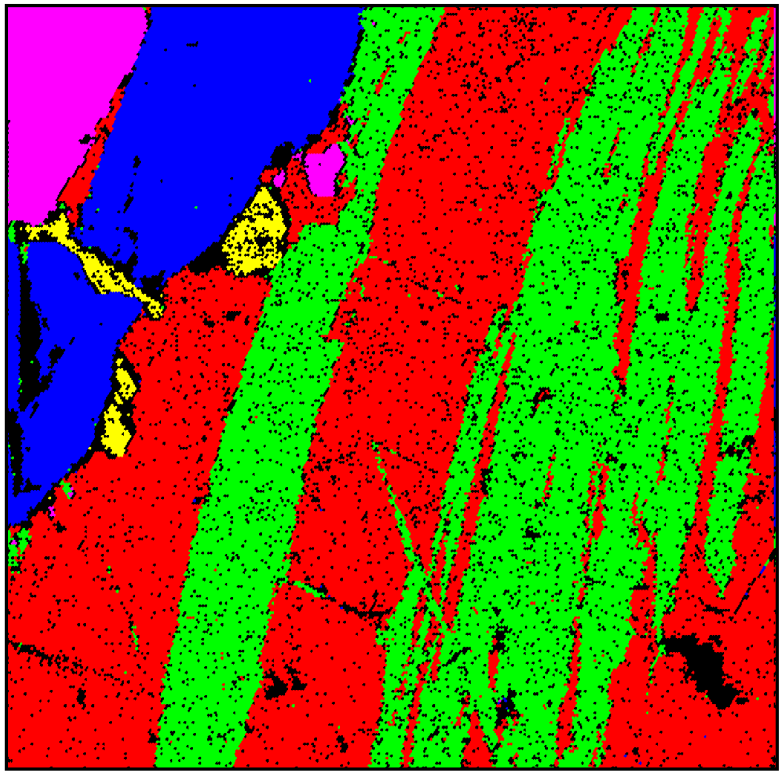

Phase	Total Fraction	Partition Fraction
Chalcopyrite	0.395	0.445
Cubanite	0.330	0.372
Amphibole	0.010	0.011
Iron Oxide	0.115	0.130
Iron Sulfide	0.039	0.044

Image quality based on Band Contrast (left) and Phase Map (right)

Courtesy of EDAX

EBSD mapping can be used to identify coesite-quartz inclusions within garnet and provide unique data about the deformation of the surrounding garnet

Sample preparation: Final polish with colloidal silica, uncoated
SEM type: FEG SEM
EBSD System: HKL CHANNEL5
Accelerating voltage: 20 kV
Probe Current: 12 nA

HKL Technology EBSD Application Catalogue 2005

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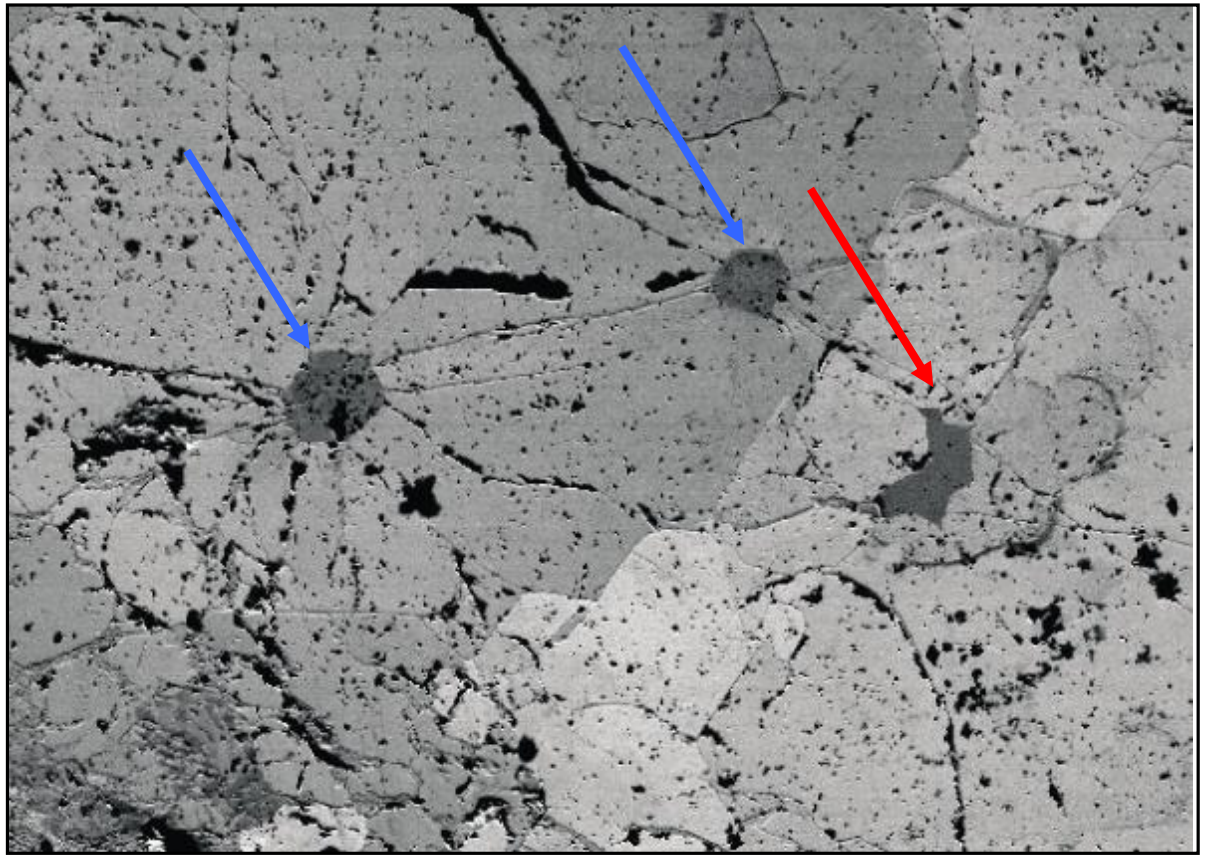

Forward scattered electron image

General microstructure of the analyzed area.

Three prominent inclusions in dark grey, two of which shows the coesite-quartz transformation (radial fractures visible as dark lines).

Grey scale variations in the surrounding garnet represent differences in crystal orientations („channeling contrast")

Field of view = 1.2 mm

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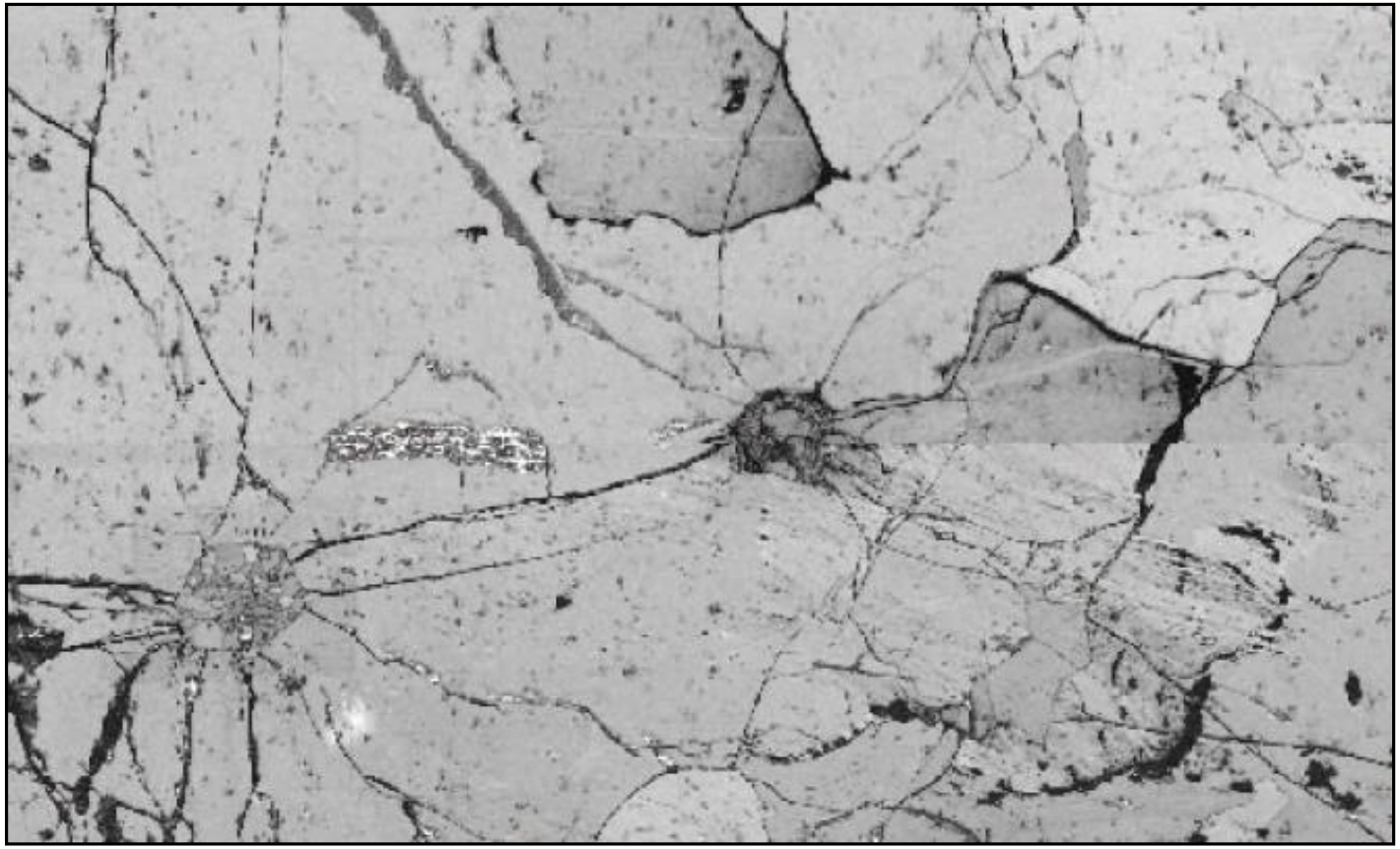

EBSD quality map

General microstructure of the analyzed area

Two of the inclusions (in the center and lower left part of the image) are prominent with radiating fractures, visible as black lines (poor EBSD quality)

Horizontal join of the 2 maps is visible across the center of the image.

Scale bar = 400µm

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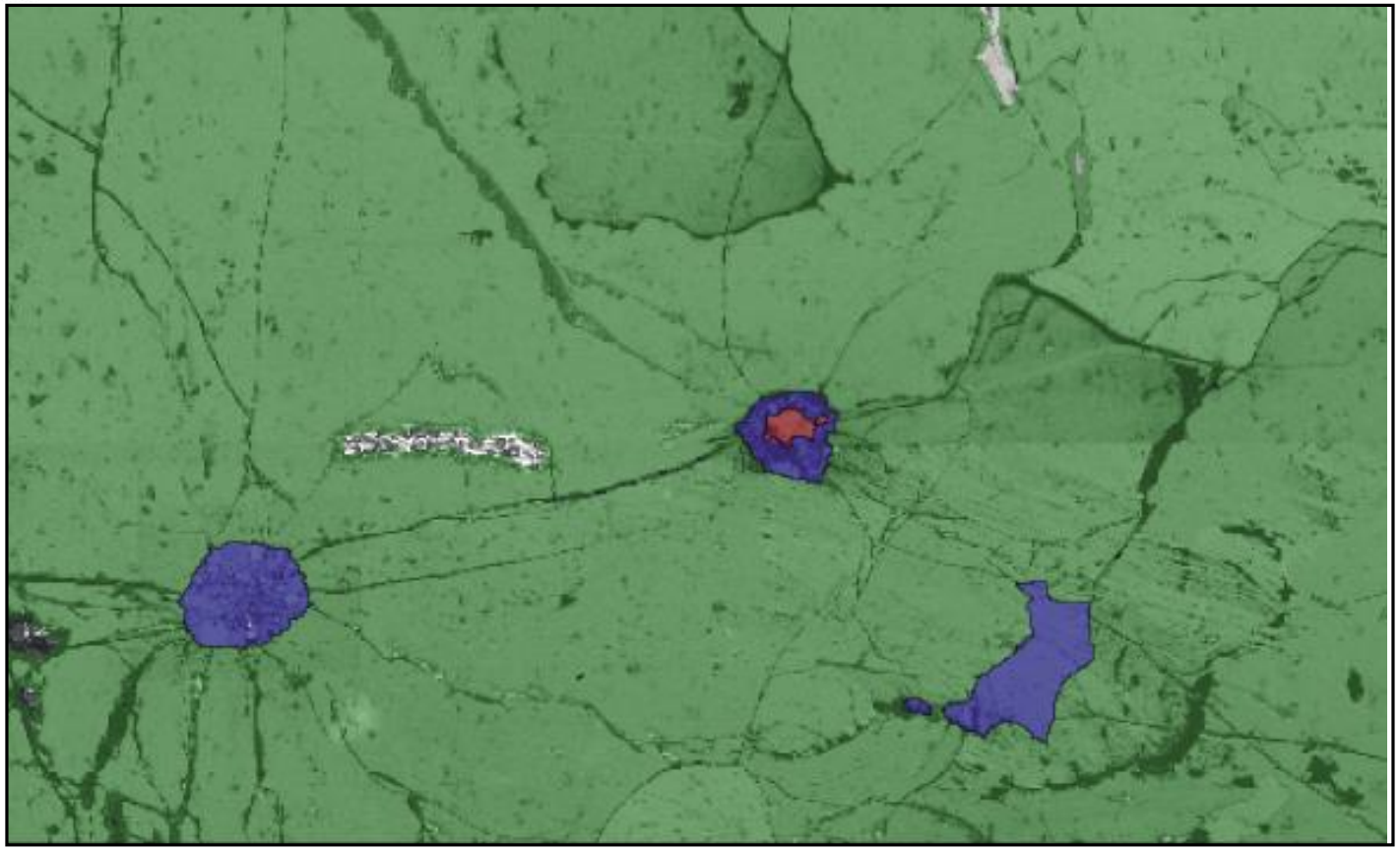

Phase map

Distribution of three phases across the area

Garnet is marked in green, quartz in blue and coesite in red.

The central inclusion has a core of coesite surrounded by a rim of quartz.

The lower-left inclusion has completely reverted to quartz.

Radiating from the prominent inclusions are fractures, visible as black lines (poor EBSD quality)

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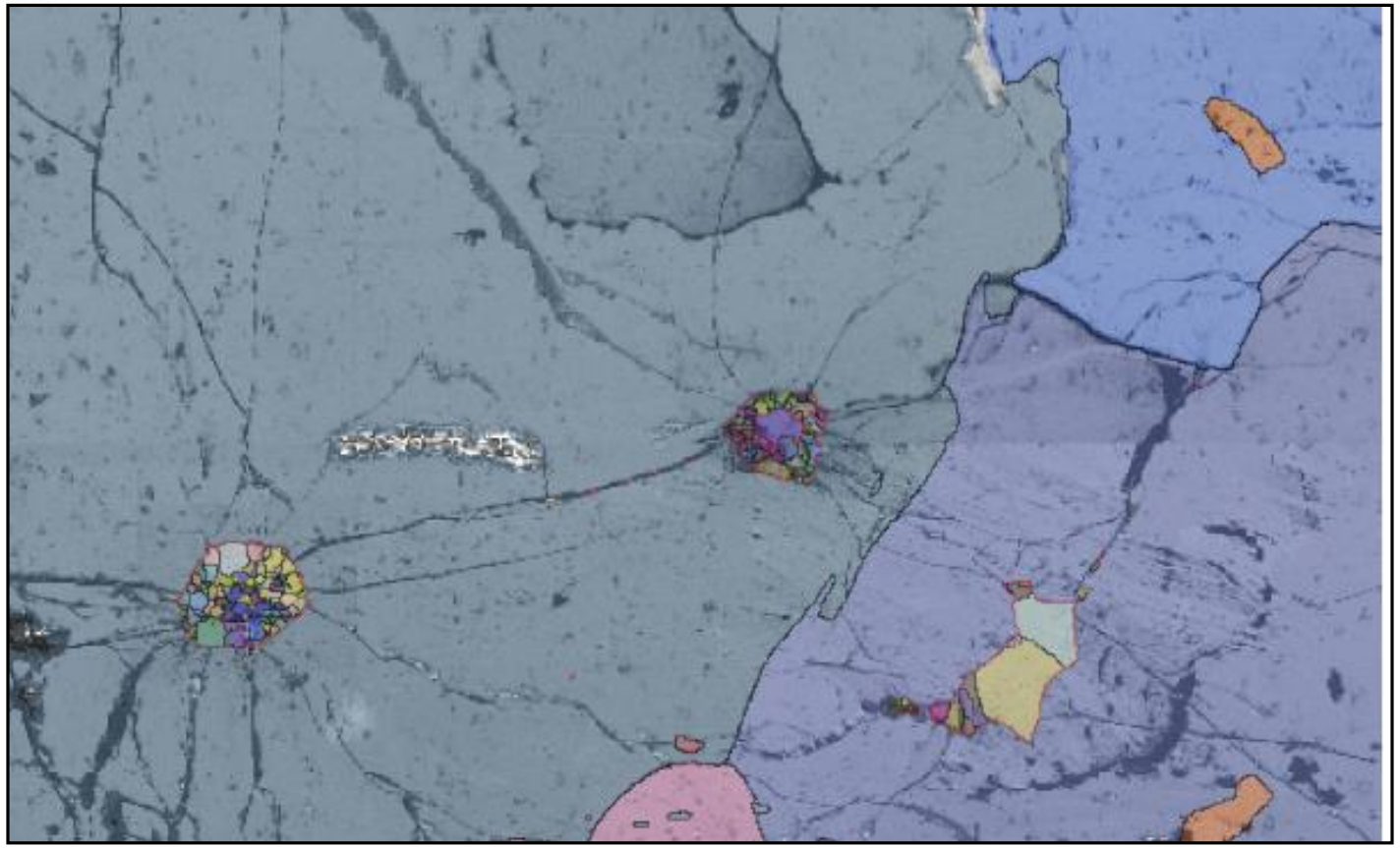

Orientation map

Crystallographic orientations of all three minerals

Colors corresponding to the Euler angles.

Grain boundaries are marked in black, phase boundaries are marked in red

The whole area is comprised of only a few garnet grains. The quartz in the inclusions is polycrystalline with grain boundaries radiating from the center. The coesite in the central inclusion is a single crystal

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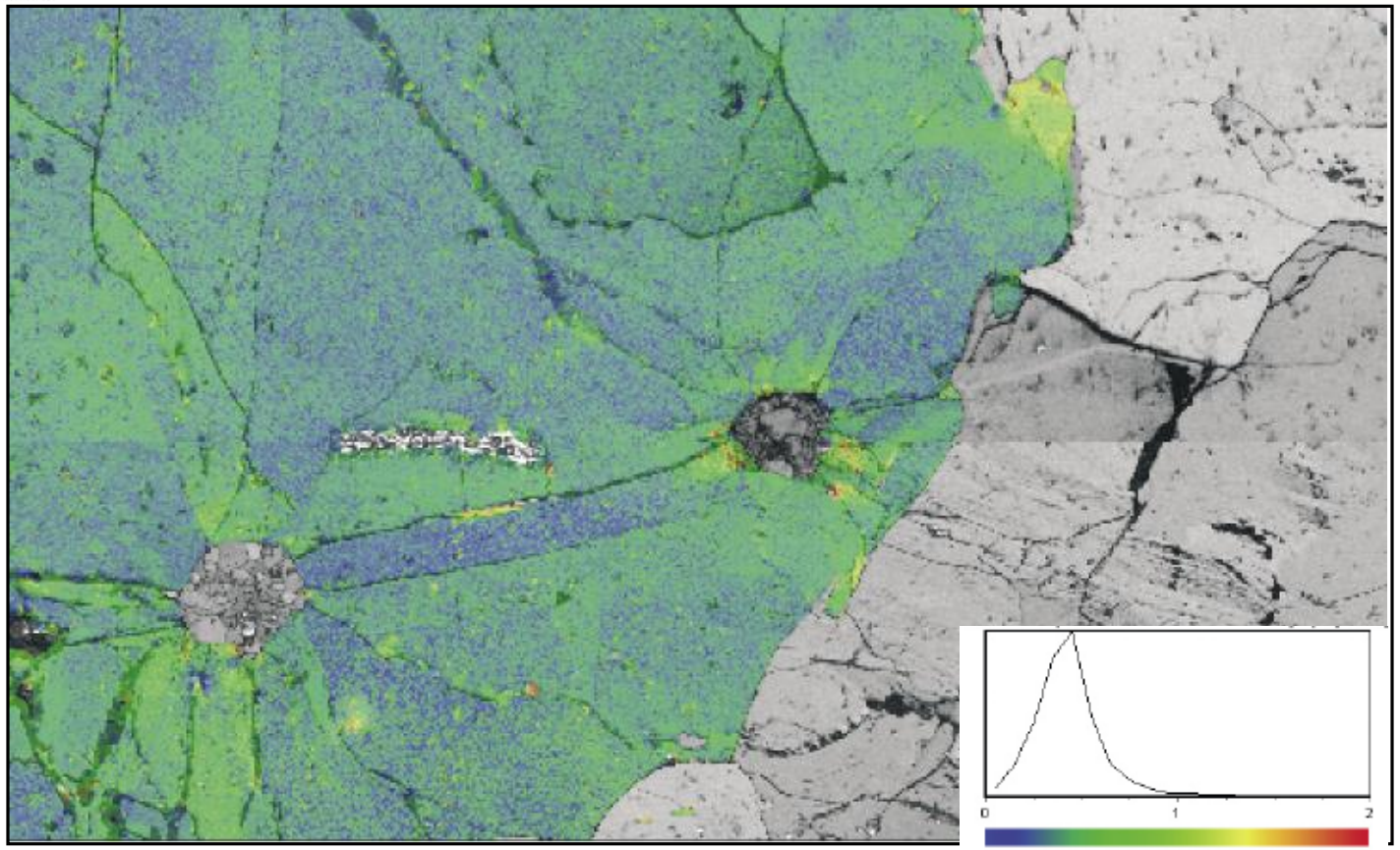

Deformation map

Deformation of the garnet around the coesite/quartz inclusions

The attempted change from coesite to quartz has tried to expand the inclusion, thereby causing radial cracks and deformation in the garnet.

This means that the garnet crystal has acted as a protective pressure vessel, so that pieces of coesite have been preserved.

Small scale of deformation in the large garnet grain (less than 2^{o})

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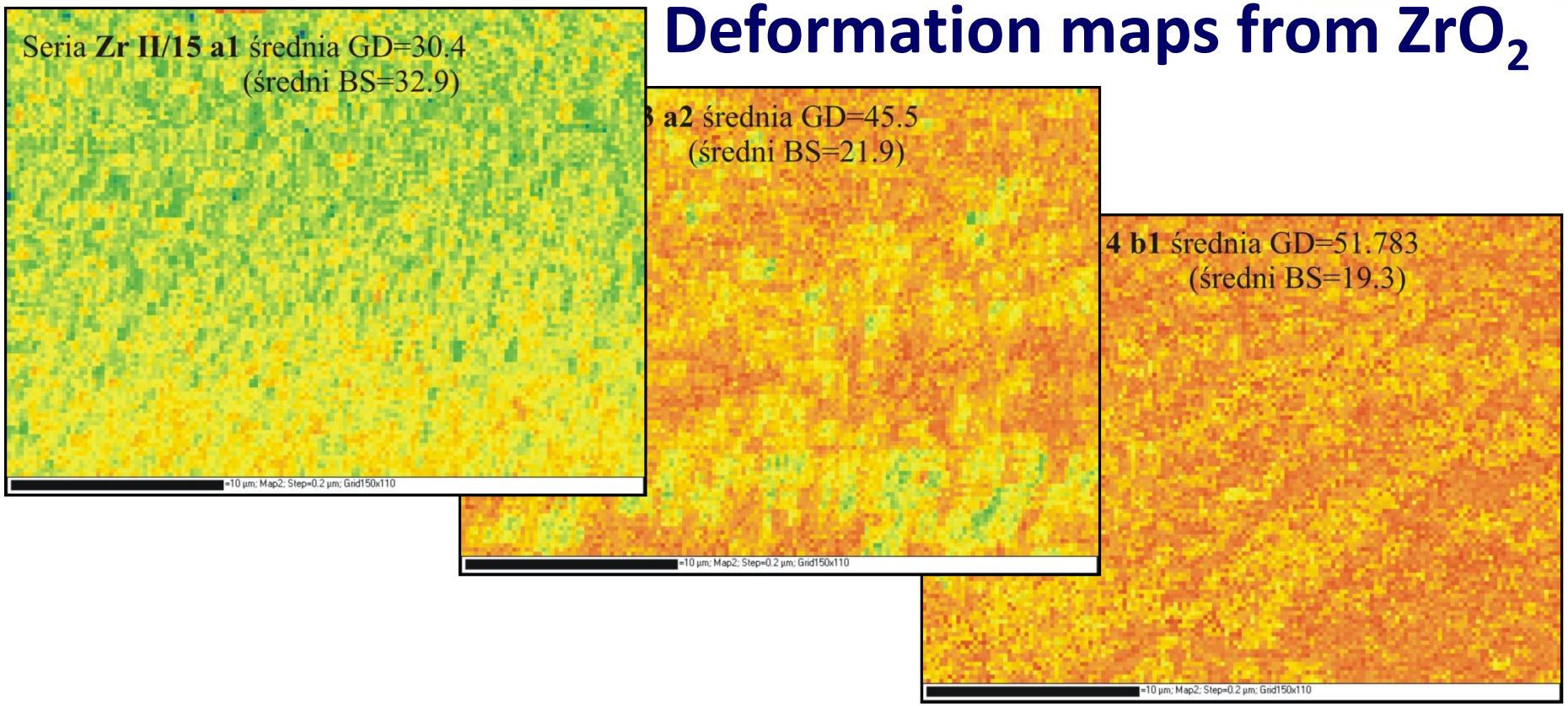
Deformation maps from ZrO_2



Development of deformation in the zirconia polycrystals exposed to „shot peening”

Deformation maps exhibit the increase of dislocation density.

Shot peening is a process used to produce a compressive residual stress layer and modify mechanical properties of surface layers in metals and ceramics by impacting a surface with shot (round metallic, glass or ceramic particles) with force sufficient to create plastic deformation.

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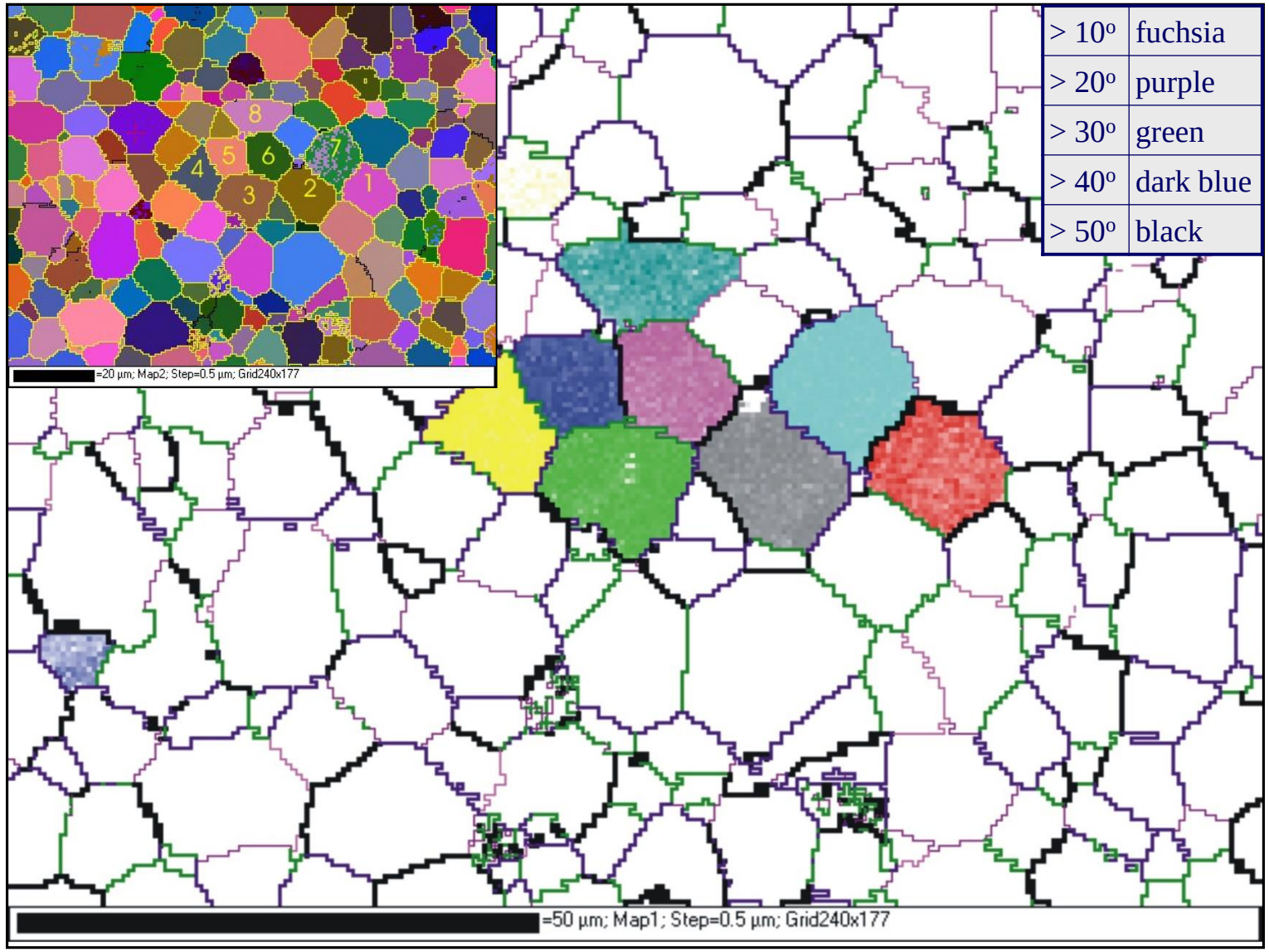

> 10°	fuchsia
> 20°	purple
> 30°	green
> 40°	dark blue
> 50°	black

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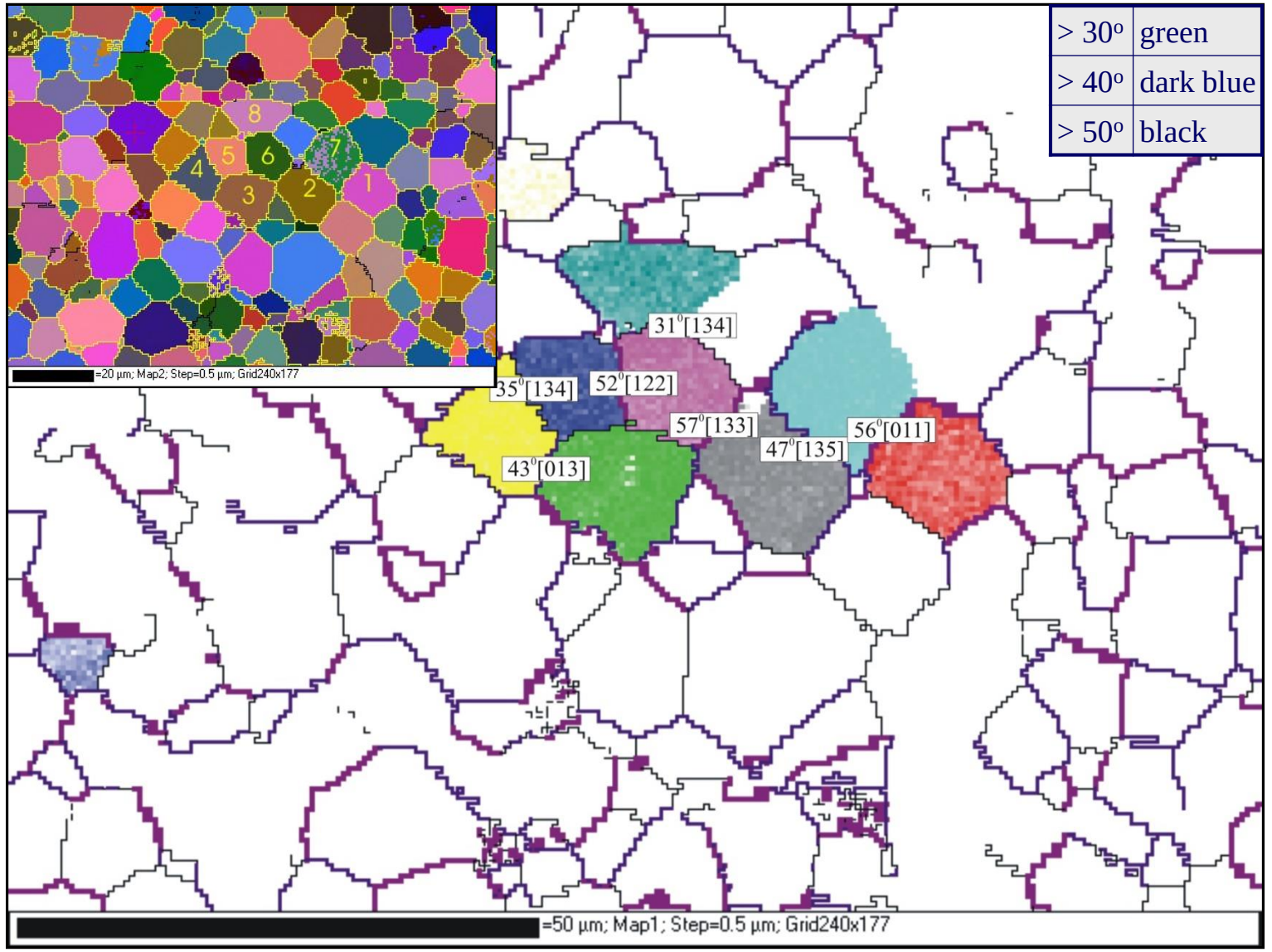

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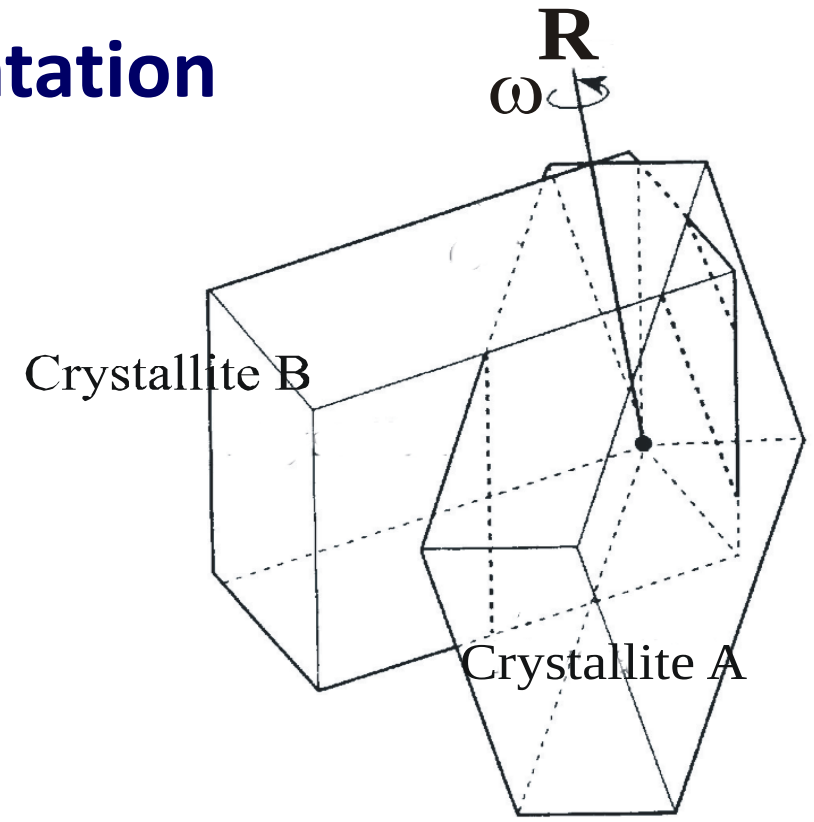
Misorientation



K. Sztwiertnia, M. Faryna, G. Sawina, Journal of Journal of Microscopy, Vol. 224, 2006, 4

Misorientation between two crystals A and B is defined as rotation transforming the crystallite B reference system into the crystallite A reference system.

$$K_A = \Gamma_{AB} \cdot K_B$$

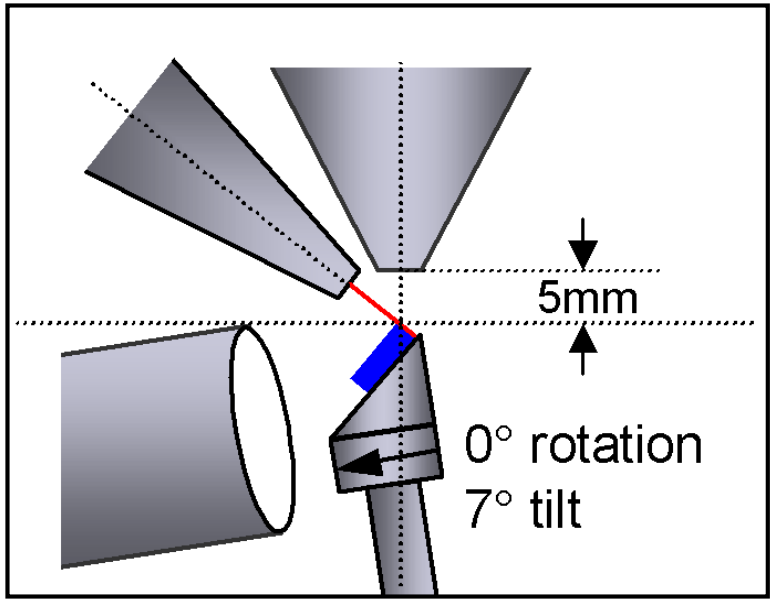
$$\Gamma^e_{AB} = S_i \cdot \Gamma_{AB} \cdot P_j \quad (i = 1,\ldots,M,\ j = 1,\ldots,N)$$

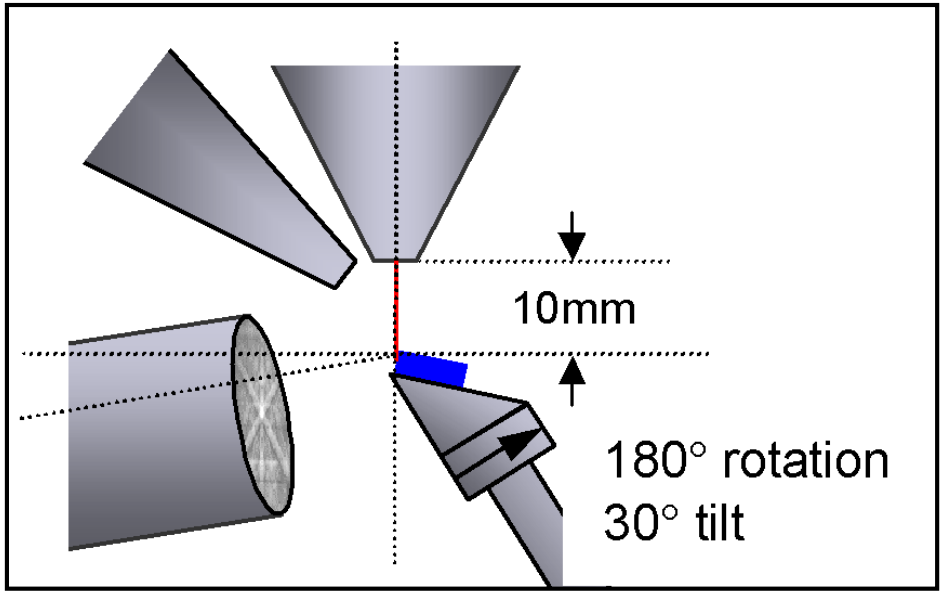
where: S_i and P_j are symmetry elements of the first and second crystal

Misorientation can be described mathematically in several ways:

- axis/angle pair
- rotation matrix,
- Euler angles,
- quaternions,
- Rodrigues vectors

Serial Sectioning in a Dual Beam FIB-SEM





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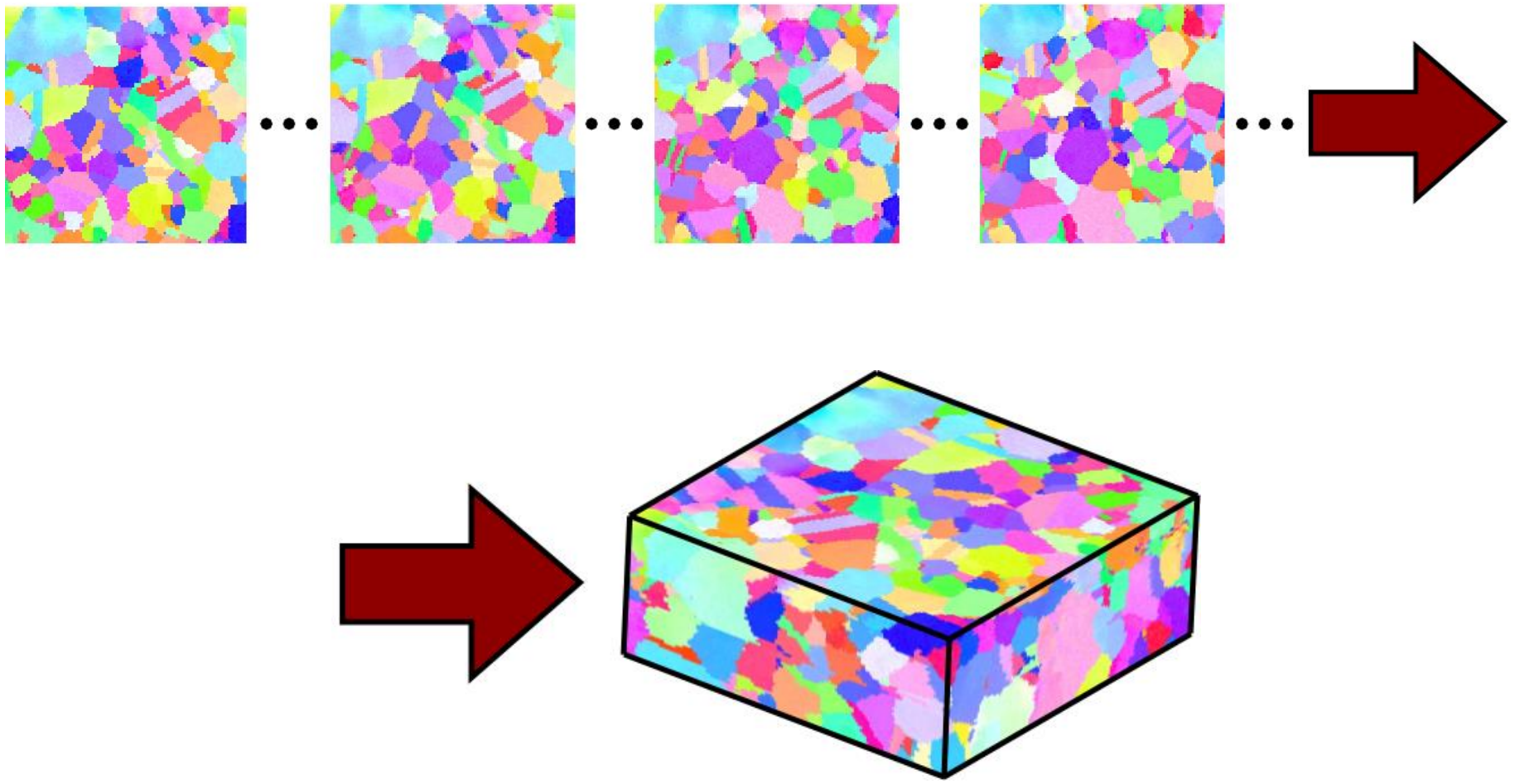
Serial Sectioning – Volume View



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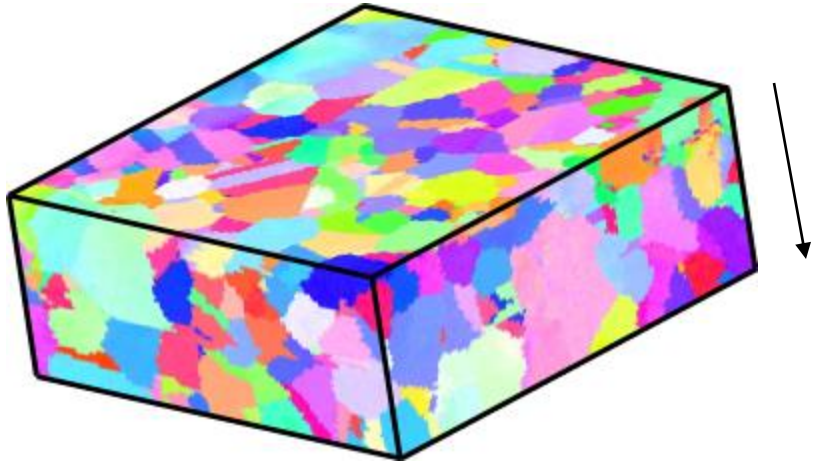
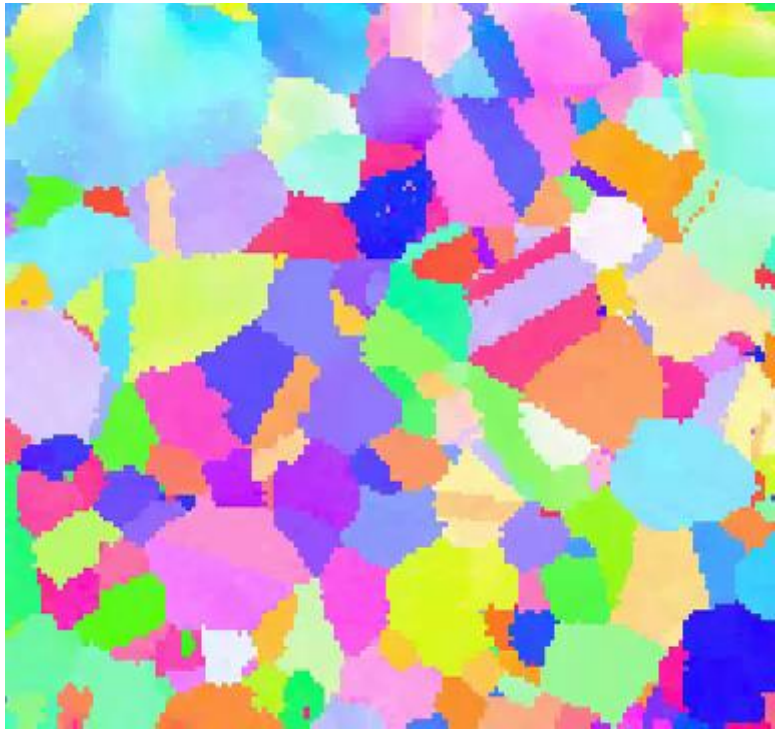
Serial Sectioning – Slices





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6. Conclusions

1. A scanning electron microscope equipped with an EBSD system enables the quantitative (!) analysis of the microstructure of crystalline materials, not only metals and alloys but also non-conductive samples.
2. Phase analysis of individual grains is possible.
3. Deformation processes can be successfully studied.
4. With a field emission scanning microscope (FEGSEM), it is possible to carry out quantitative analyses of grains/subgrains as small as ~100 nm, depending on the type of material.

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5. Maps of crystal orientation can be collected using EBSD. They remove any ambiguity regarding the recognition of grains and grain boundaries in the sample.
6. The grains in polycrystalline material are usually not randomly oriented and crystallographic texturing can confer special properties on materials. Thus, EBSD is as an important technique for texture analysis allowing the relation between texture and microstructure to be studied.
7. Boundaries formed between grains with particular orientation relationships to one another can have desirable properties. EBSD can characterize these boundaries and measure the distribution of various boundary types in a sample.